













STRUCTURE REPORTS

for 1982

Volume 49B

Part 1

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# STRUCTURE REPORTS

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Volume 49B (Part 1)

ORGANIC SECTION

*General editor*

G. Ferguson

*Section editor*

G. Ferguson

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## INTRODUCTION

The present volume continues the aim of Structure Reports to present critical accounts of all crystallographic structure determinations. Details of the arrangement in the volumes, symbols used etc. are given in previous volumes (e.g. 41B or 42A, pages vi-viii).

University of Guelph,  
Guelph, Ontario, Canada

G. FERGUSON

July 1st, 1989



# STRUCTURE REPORTS

## SECTION III

# ORGANIC COMPOUNDS

*Edited by*

G. Ferguson

*with the assistance of*

M. Currie

D. L. Hughes

C. H. L. Kennard

A. L. Macdonald

W. C. Marsh

D. F. Rendle

S. J. Rettig

S. N. Scrimgeour

To find a particular organic or organometallic compound the subject index or formula index at the end of Part 2 of this volume should be used. The general arrangement is: aliphatic or open-chain compounds; open chains with N, S; benzene derivatives; cyclic hydrocarbons; condensed ring systems; heterocyclic compounds; carbohydrates; amino acids; natural products; molecular complexes; organometallic compounds - B, Si, P, As, Sb, groups IA, IIA, III, IV, VI; transition metal complexes -  $\pi$ -complexes, other ligands; inorganic anions which have organic counterions. Only complete structure analyses are described, and those which have been reported in preliminary communications and for which details will appear at a later date, have not been described here.

## POTASSIUM FLUORIDE MALONIC ACID



J. EMSLEY, D.J. JONES and R. KURODA, 1982. J. Chem. Soc. Dalton, 1179-1184.

Monoclinic, Cc,  $a = 19.463$ ,  $b = 18.920$ ,  $c = 8.735$  Å,  $\beta = 112.5^\circ$ ,  $Z = 20$ . Mo radiation,  $R = 0.116$  for 4708 reflexions. [The crystals deteriorate in air.]

The crystals, which are unstable with evolution of HF, show a remarkable cleavage. The X-ray analysis reveals a polymeric crystal arrangement with alternate malonic acid molecules and fluoride ions. There are five distinct malonic-acid-fluoride chains in the unit cell held together by hydrogen bonds ( $\text{O}\cdots\text{F}$  2.41-2.49(4) Å). The potassium ions have an environment of two F atoms (K-F 2.584-2.738) and six O atoms (K-O 2.797-2.962 Å) (Fig. 1). The IR spectra are discussed in detail.

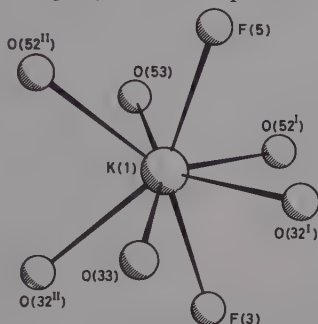
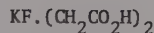


Fig. 1.  $\text{C}_3\text{H}_4\text{FKO}_4$ : the potassium ion environment.

## POTASSIUM FLUORIDE SUCCINIC ACID



J. EMSLEY, D.J. JONES, R.S. OSBORN and R.E. OVERILL, 1982. J. Chem. Soc. Dalton, 809-813.

Orthorhombic, Pnam,  $a = 7.068$ ,  $b = 5.571$ ,  $c = 16.846$  Å,  $Z = 4$ . Cu radiation,  $R = 0.035$  for 637 reflexions.

The structure (Fig. 1) consists of infinite chains of  $\text{F}^-$  ions joined to two succinic acid molecules by very short hydrogen bonds ( $\text{O}\cdots\text{F}$  2.441(3) Å). Each  $\text{K}^+$  ion has two F atoms (2.684, 2.874(3)) and four carbonyl O atoms as nearest neighbours (K-O 2.818-2.843(3) Å). Distances within the acid group are: H(1)-O(1) 0.85(3), C(1)-O(1) 1.305(3), C(1)-O(2) 1.211(3), C(1)-C(2) 1.509(3), C(2)-H(21) 1.03(3), C(2)-H(22) 0.95(3), C(2)-C(2') 1.514(5) Å.

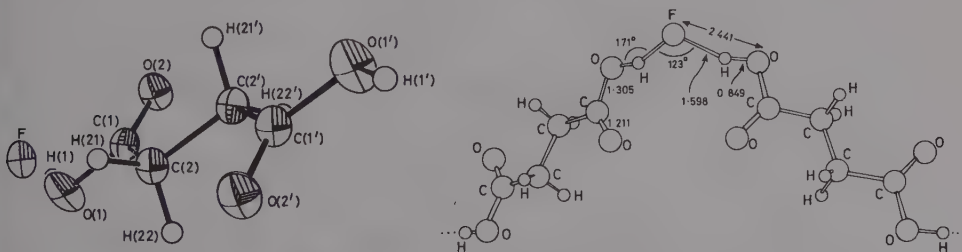
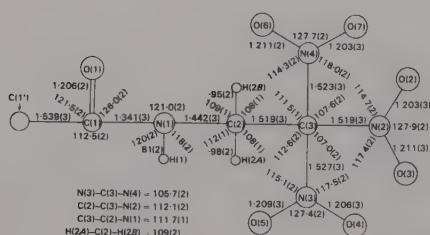


Fig. 1.  $\text{C}_4\text{H}_6\text{FKO}_4$ : the molecule and the hydrogen-bonded environment around the  $\text{F}^-$  ion.

$$\text{C}_{66}\text{H}_{68}\text{N}_8\text{O}_{14}$$

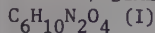
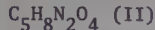
Triclinic,  $P\bar{1}$ ,  $a = 6.6574$ ,  $b = 6.6855$ ,  $c = 8.9634$  Å,  $\alpha = 86.715$ ,  $\beta = 81.263$ ,  $\gamma = 76.025^\circ$ ,  $Z = 1$ . Cu radiation,  $R = 0.034$  for 1151 reflexions.

$$(O_2N)_3C-CH_2-NH-\overset{\overset{O}{\parallel}}{C}-\overset{\overset{O}{\parallel}}{C}-NH-CH_2-C(NO_2)_3$$

$$\text{C}_{66}\text{H}_{60}\text{O}_7, 2\text{H}_2\text{O}$$

Triclinic,  $P\bar{1}$ ,  $a = 7.219$ ,  $b = 8.290$ ,  $c = 8.693$  Å,  $\alpha = 66.97$ ,  $\beta = 88.41$ ,  $\gamma = 82.12^\circ$ ,  $D_m = 1.58$ ,  $Z = 2$ . Mo radiation,  $R = 0.032$  for 1399 reflexions.

Fig. 1. The  $C_6H_6O_7 \cdot 2H_2O$  structure.



METHYL  $\beta$ -DIMETHYLAMINO- $\alpha$ -NITROACRYLATEMETHYL  $\beta$ -METHYLAMINO- $\alpha$ -NITROACRYLATE

V.G. ANDRIANOV, Yu.T. STRUCHKOV and K.K. BABIEVSKY, 1982. Cryst. Struct. Comm., 11, 35-38.

I. Triclinic,  $P\bar{1}$ ,  $a = 7.208$ ,  $b = 7.466$ ,  $c = 8.752$  Å,  $\alpha = 92.40^\circ$ ,  $\beta = 104.02^\circ$ ,  $\gamma = 109.92^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.054$  for 809 reflexions.

II. Monoclinic,  $P2_1/a$ ,  $a = 6.336$ ,  $b = 11.034$ ,  $c = 10.095$  Å,  $\beta = 92.45^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.112$  for 647 reflexions.

Molecule I has trans-geometry, whereas II is cis (Fig. 1). There are distortions attributable to molecular overcrowding, as shown in the figures.

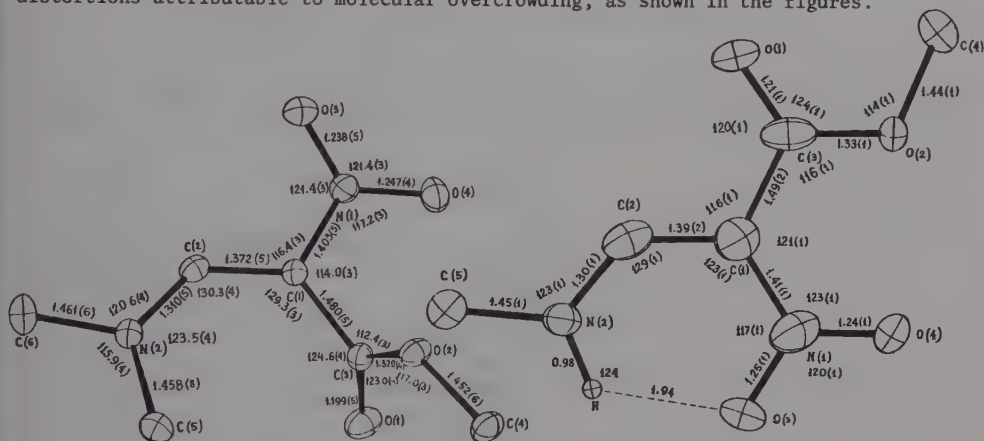
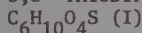
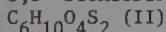


Fig. 1. The methyl esters  $C_6H_{10}N_2O_4$  (left) and  $C_5H_8N_2O_4$  (right).

## 3,3'-THIODIPROPIONIC ACID



## 3,3'-DITHIODIPROPIONIC ACID



K. PROUT and S. HERNÁNDEZ-CASSOU, 1982. Acta Cryst., B38, 338-340.

I. Orthorhombic,  $Pcan$ ,  $a = 5.063$ ,  $b = 8.648$ ,  $c = 18.073$  Å,  $Z = 4$ . Mo radiation,  $R = 0.038$  for 523 reflexions.

II. Monoclinic,  $C2/c$ ,  $a = 5.466$ ,  $b = 8.870$ ,  $c = 18.906$  Å,  $\beta = 95.99^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.034$  for 626 reflexions. [See also (1).]

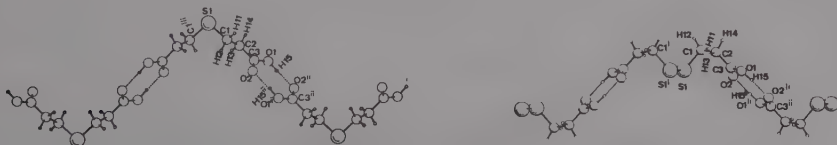


Fig. 1. Thiodipropionic acid (I) (left) and dithiodipropionic acid (II) (right); the twofold axis is located at the S atom (I) or at the middle of the S-S bond (II).

Both structures (Fig. 1) consist of infinite chains of molecules linked by intermolecular hydrogen bonds with O...O 2.674 Å (I) and 2.658 Å (II); the carboxylic acid groups and associated hydrogen bonds are coplanar, but not coplanar with the remaining non-hydrogen atoms of the molecule. Bond lengths and angles are generally as expected with S-C 1.818 (I) and 1.815 Å (II) and S-S 2.032 Å (II).

1. This Volume, p6.

# ETHYLENEDITHIODIACETIC ACID



L.M. SHKOL'NIKOVA, T.N. SAFONOVA, M.A. PORAI-KOSHITS, N.M. DYATLOVA and J. PODLAHA, 1982. Zh. Strukt. Khim., 23-4, 96-100 [J. Struct. Chem., 23, 569-573].

Triclinic,  $P\bar{1}$ ,  $a = 5.100$ ,  $b = 10.005$ ,  $c = 10.109$  Å,  $\alpha = 116.65$ ,  $\beta = 92.71$ ,  $\gamma = 95.91^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.031$  for 2547 reflexions.

The two crystallographically independent centrosymmetric molecules (Fig. 1) have different configurations. O-H...O hydrogen bonding results in layers of molecules parallel to the (110) plane. Bond lengths and angles ( $\sigma = 0.003$ - $0.006$  Å and  $0.1$ - $0.3^\circ$ ) are normal.

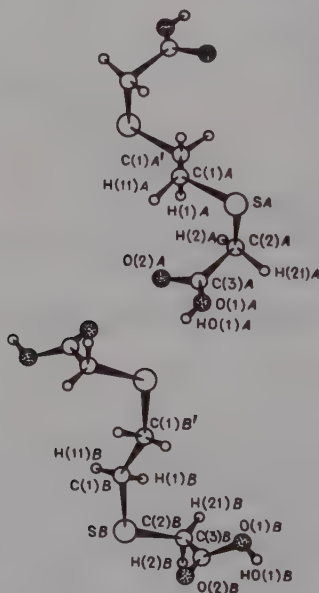
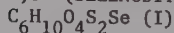
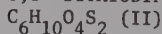


Fig. 1. The two independent  $\text{C}_6\text{H}_{10}\text{O}_4\text{S}_2$  molecules.

# 3,3'-(SELENODITHIO)DIPROPIONIC ACID



# 3,3'-DITHIODIPROPIONIC ACID



G.V.N. APPA RAO, M. SESHASAYEE, G. ARAVAMUDAN, T.N. RAO and P.N. VENKATASUBRAMANIAN, 1982. Acta Cryst., B38, 2852-2855.

I. Monoclinic,  $C2/c$ ,  $a = 23.283$ ,  $b = 9.027$ ,  $c = 4.997$  Å,  $\beta = 97.09^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.073$  for 773 reflexions.

II. Monoclinic,  $C2/c$ ,  $a = 5.4751$ ,  $b = 8.8691$ ,  $c = 18.930$  Å,  $\beta = 95.800^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.031$  for 673 reflexions. [See also the preceding Report.]

In compound I (Fig. 1) the Se atom, which is located on a diad axis, is bonded to two S atoms with Se-S 2.180(3) Å and S-Se-S 105.0(2)°. Compound II also has twofold symmetry and the S-S and S-C bond lengths are 2.033(1) and 1.819(3) Å respectively. In both crystal structures the molecules are linked into chains by O-H...O hydrogen bonds, with O...O 2.681 Å in I and 2.662 Å in II.

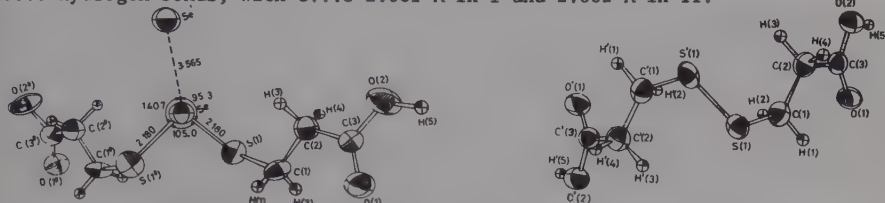


Fig. 1. Perspective views of molecules of  $\text{Se}(\text{SCH}_2\text{CH}_2\text{COOH})_2$  (I) and  $(\text{SCH}_2\text{CH}_2\text{COOH})_2$  (II).

1. This Volume, p5.

2-(CYANOMETHYL)-2-METHYL-3-OXOBUTANAMIDE

$\text{C}_7\text{H}_{10}\text{N}_2\text{O}_2$

$\text{CH}_3\text{-CO-C}(\text{CH}_3)(\text{CH}_2\text{CN})\text{-CO-NH}_2$

R. SCHWESINGER, R. WADITSCHATKA, J. RIGBY, R. NORDMANN, W.B. SCHWEIZER, E. ZASS and E. ESCHENMOSER, 1982. *Helv. Chim. Acta*, **65**, 600-610.

Monoclinic,  $P2_1/n$ ,  $a = 7.091$ ,  $b = 13.335$ ,  $c = 9.436$  Å,  $\beta = 113.24^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.04$  for 983 reflexions.

The analysis of this 3-oxobutanamide provided proof-of-structure for this intermediate.

(4-CHLOROPHENOXY)ACETIC ACID

$\text{C}_8\text{H}_7\text{ClO}_3$

S.V. KUMAR and L.M. RAO, 1982. *Acta Cryst.*, **B38**, 2062-2064.

Triclinic,  $P\bar{1}$ ,  $a = 7.108$ ,  $b = 7.407$ ,  $c = 8.1217$  Å,  $\alpha = 88.11$ ,  $\beta = 108.24$ ,  $\gamma = 94.05^\circ$ ,  $D_m = 1.525$ ,  $Z = 2$ . Cu radiation,  $R = 0.059$  for 819 reflexions.

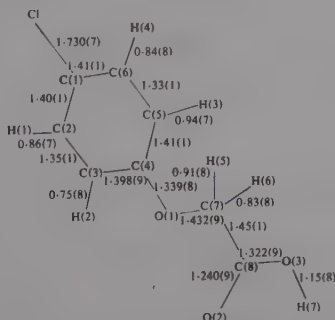


Fig. 1. Bond lengths in (4-chlorophenoxy)acetic acid.

The molecules (Fig. 1) are linked into dimers across a centre of symmetry by pairs of hydrogen bonds ( $\text{O}\cdots\text{O}$  2.610(7) Å), and are stacked in layers parallel to (010). Except for C(7)-C(8), bond lengths compare well with those from an earlier

study of the same molecule (I). There is a short, intermolecular  $\text{Cl}\cdots\text{Cl}$  distance ( $3.349(3)$  Å) with  $\text{C}(1)-\text{Cl}\cdots\text{Cl}'$   $177.1(3)^\circ$ . The planar acetic acid group makes an angle of  $3.2(3)^\circ$  with the ring plane.

# 1. Structure Reports, 48B, 112.

METHYL  $\beta$ -NITROCINNAMATE

$\text{C}_{10}\text{H}_9\text{NO}_4$  (I)

METHYL  $\alpha$ -(3-INDOLYL)-NITROACRYLATE

$\text{C}_{12}\text{H}_{10}\text{N}_2\text{O}_4$  (II)

V.G. ANDRIANOV, Yu.T. STRUCHKOV and K.K. BABIEVSKY, 1982. Cryst. Struct. Comm., 11, 31-34.

I. Triclinic,  $P\bar{1}$ ,  $a = 9.828$ ,  $b = 10.297$ ,  $c = 11.607$  Å,  $\alpha = 67.71$ ,  $\beta = 67.29$ ,  $\gamma = 70.56^\circ$ ,  $D_m = 1.30$ ,  $Z = 4$ . Mo radiation,  $R = 0.042$  for 1963 reflexions.

II. Monoclinic,  $P2_1/m$ ,  $a = 7.951$ ,  $b = 6.679$ ,  $c = 10.572$  Å,  $\beta = 94.17^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.057$  for 1207 reflexions.

There are two independent molecules of I in the asymmetric unit; molecule II lies on a crystallographic mirror plane. All the molecules (Fig. 1) have the Z configuration.

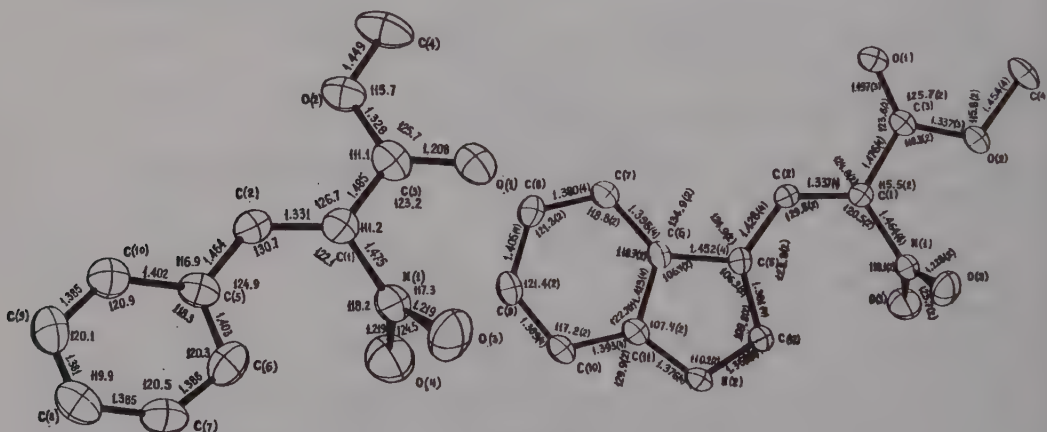


Fig. 1. Views of the methyl esters  $\text{C}_{10}\text{H}_9\text{NO}_4$  (left) and  $\text{C}_{12}\text{H}_{10}\text{N}_2\text{O}_4$  (right).

$\alpha$ -METHYL-trans-CINNAMIC ACID

$\text{C}_{10}\text{H}_{10}\text{O}_2$

$\text{C}_6\text{H}_5\text{CH}=\text{C}(\text{CH}_3)\text{CO}_2\text{H}$

R.F. BRYAN and D.H. WHITE, 1982. Acta Cryst., B38, 1332-1334.

Monoclinic,  $P2_1/c$ ,  $a = 7.633$ ,  $b = 11.454$ ,  $c = 11.514$  Å,  $\beta = 120.91^\circ$ ,  $D_m = 1.24$ ,  $Z = 4$ . Cu radiation,  $R = 0.041$  for 812 reflexions.

The molecules are present in the crystal as centrosymmetric hydrogen-bonded dimers ( $\text{O-H}\cdots\text{O}$   $2.625$  Å) (Fig. 1). The molecule is closely planar overall: the plane of the phenyl ring is inclined at  $3.4^\circ$  to that of the double bond which, in turn, is inclined by a further  $4.3^\circ$  to the plane of the carboxy group.

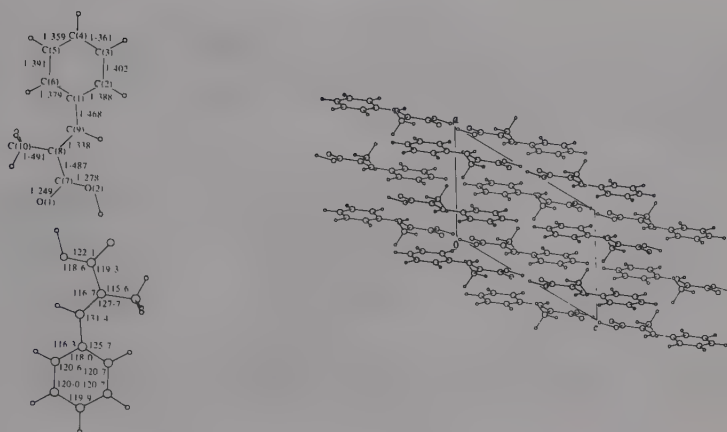
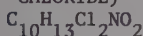


Fig. 1.  $C_{10}H_{10}O_2$ : projection on to the dimer plane showing bond lengths and angles (left) and a view of the molecular packing in b-axis projection (right).

(R)- $\gamma$ -AMINO- $\beta$ -(p-CHLOROPHENYL)-BUTYRIC ACID MONOHYDROCHLORIDE ((R)-BACLOFEN MONOHYDROCHLORIDE)



C.-H. CHANG, D.S.C. YANG, S.C. YOO, B.-C. WANG, J. PLETCHER, M. SAX and C.F. TERRENCE, 1982. *Acta Cryst.*, B38, 2065-2067.

Orthorhombic,  $P2_12_12_1$ ,  $a = 6.373$ ,  $b = 7.318$ ,  $c = 25.699$  Å,  $Z = 4$ . Cu radiation,  $R = 0.029$  for 1169 reflexions.

The molecules (Fig. 1) are linked into infinite chains along  $b$  by hydrogen bonding ( $O(1)-H \dots Cl$  3.069(3) and  $O(2) \dots H(3)N-N$  2.845(3) Å. Each amino H atom is also involved in hydrogen bonds to  $Cl^-$  ( $N-H \dots Cl$  3.196, 3.246 and 3.272(3) Å). Displacements from the ring plane are:  $Cl$  -0.053 and  $C(3)$  -0.178 Å. The  $C(4)-N$  bond length is 1.499(4) Å.

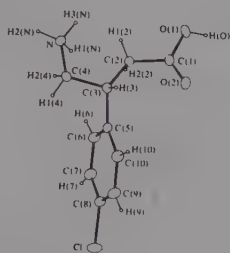
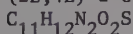


Fig. 1. The (R)-baclofen cation.

(2E,4E)-2-CYANO-4-METHYL-5-THIOCYANATO-2,4-HEXADIENOIC ACID ETHYL ESTER



P. KNUUTTILA, H. KNUUTTILA, B. SCHULZE, M. MÜHLSTÄDT and J. KAISER, 1982. *Acta Cryst.*, B38, 335-337.

Monoclinic,  $P2_1/c$ ,  $a = 4.551$ ,  $b = 19.846$ ,  $c = 13.358$  Å,  $\beta = 90.49^\circ$ ,  $D_m = 1.32$ ,  $Z = 4$ . Mo radiation,  $R = 0.046$  for 1178 reflexions.



The main feature of the structure (Fig. 1) is the presence of a substituted (E)-s-trans-(E)-1,3-butadiene. Bond lengths are as expected, except for a shortening of C(3)-C(4) to 1.443 Å, and bond angles are characteristic of tetrahedral and trigonal bonding, though the C(2)-C(3)-C(4) angle is widened to 132.4° by steric interaction of the cyanide and methyl groups.

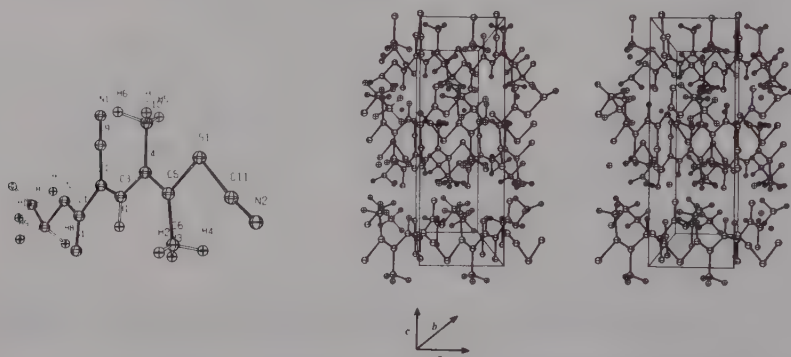
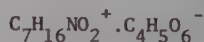


Fig. 1. The  $C_{11}H_{12}N_2O_2S$  molecule (left) and a stereoscopic drawing of the unit cell (right).

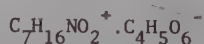
ACETYLCHOLINE HYDROGEN ( $\pm$ )-TARTRATE

$C_{11}H_{21}NO_8$  (I)



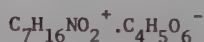
ACETYLCHOLINE HYDROGEN (+)-TARTRATE (POLYMORPH 1)

$C_{11}H_{21}NO_8$  (II)



ACETYLCHOLINE HYDROGEN (+)-TARTRATE (POLYMORPH 2)

$C_{11}H_{21}NO_8$  (III)



B. JENSEN, 1982. *Acta Cryst.*, **B38**, 1185-1192.

I. Orthorhombic,  $Pna2_1$ ,  $a = 11.853$ ,  $b = 8.615$ ,  $c = 14.111$  Å,  $D_m = 1.37$ ,  $Z = 4$ . Mo radiation,  $R = 0.035$  for 1950 reflexions.

II. Monoclinic,  $P2_1$ ,  $a = 6.198$ ,  $b = 14.530$ ,  $c = 8.043$  Å,  $\beta = 97.76^\circ$ ,  $D_m = 1.37$ ,  $Z = 2$ . Mo radiation,  $R = 0.030$  for 1426 reflexions.

III. Monoclinic,  $P2_1$ ,  $a = 12.323$ ,  $b = 8.518$ ,  $c = 13.453$  Å,  $\beta = 90.45^\circ$ ,  $D_m = 1.39$ ,  $Z = 4$ . Mo radiation,  $R = 0.069$  for 1672 reflexions.

In all three structures (Fig. 1) the hydrogen tartrate ions are interlinked via hydrogen bonds into infinite chains. The acetylcholine ions are not involved in hydrogen-bond formation in any of the structures, and no distinct contacts between the formally charged groups are found. Some disorder is present in II and III.

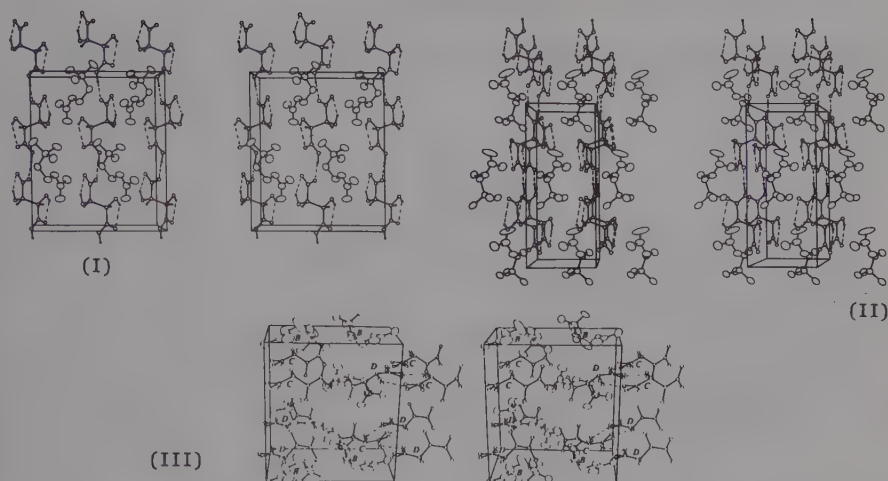
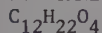


Fig. 1. Stereoscopic views of the packing of  $C_{11}H_{21}NO_8$  (I),  $C_{11}H_{21}NO_8$  (II) and  $C_{11}H_{21}NO_8$  (III).

#### DODECANEDIOIC ACID



M. VANIER and F. BRISSE, 1982. *Acta Cryst.*, **B38**, 643-645.

Monoclinic,  $P2_1/c$ ,  $a = 13.105$ ,  $b = 4.921$ ,  $c = 10.183$  Å,  $\beta = 97.40^\circ$ ,  $D_m = 1.19$ ,  $Z = 2$ . Cu radiation,  $R = 0.036$  for 548 reflexions.

The structure has been previously solved but with a different unit cell (1). The centrosymmetric molecules (Fig. 1) have mean values for  $C-C(sp^3)$  of  $1.509(5)$  Å and for  $C-C-C$  angles of  $115.1^\circ$  excluding  $C(2)-C(3)-C(4)$ . The repeat  $-CH_2-$  distance along the carbon chain is  $2.535(5)$  Å. The mean plane of  $C(1)-C(6)$  makes an angle of  $8.7^\circ$  with the  $O(1)O(2)C(1)$  plane and torsion angles along the chain are in the range  $178.2-180^\circ$ . The molecules form infinite chains linked by centrosymmetric pairs of hydrogen bonds ( $O \cdots O$   $2.654(3)$  Å).

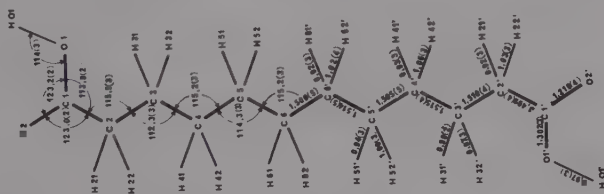
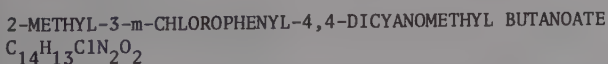


Fig. 1. The molecule of dodecanedioic acid with bond lengths and angles.

#### 1. Structure Reports, **31B**, 20.



J.H. NOORDIK, 1982. *Cryst. Struct. Comm.*, **11**, 1347-1350.

Triclinic,  $P\bar{1}$ ,  $a = 7.967$ ,  $b = 9.478$ ,  $c = 10.046$  Å,  $\alpha = 90.06$ ,  $\beta = 93.32$ ,  $\gamma = 113.13^\circ$ ,

$Z = 2$ . Mo radiation,  $R = 0.048$  for 1916 reflexions.

The configuration of the molecule is shown in Fig. 1. Relevant torsion angles are:  $C1-C2-C3-C4$   $-57.6^\circ$ ,  $C1-C2-C3-C7$   $175.4^\circ$ ,  $C13-C2-C3-C4$   $-176.8^\circ$ ,  $C13-C2-C3-C7$   $56.1^\circ$ .

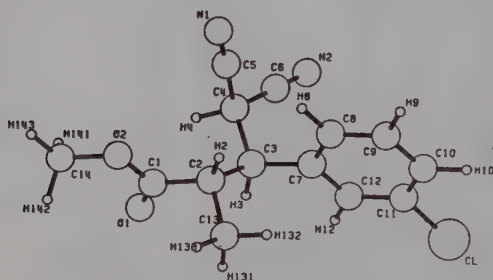


Fig. 1. The  $C_{14}H_{13}ClN_2O_2$  molecule.

2-METHYL-3-PHENYL-4,4-DICYANOMETHYLBUTANOATE  
 $C_{14}H_{14}N_2O_2$

H.M. DOESBURG, J.H. NOORDIK and P.T. BEURSKENS, 1982. *Z. Krist.*, **161**, 279-288.

Orthorhombic,  $Pbca$ ,  $a = 8.925$ ,  $b = 14.077$ ,  $c = 20.237$  Å,  $Z = 8$ . Mo radiation,  $R = 0.032$  for 458 reflexions.

A view of the molecule is given in Fig. 1. Molecular dimensions are normal. The phenyl and ester groups show a staggered conformation.

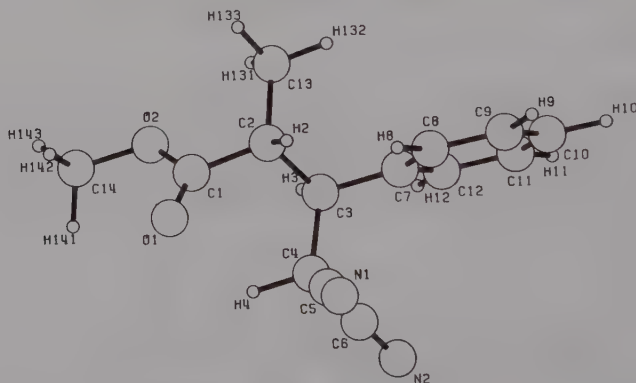


Fig. 1. A view of  $C_{14}H_{14}N_2O_2$ .

DIPHENYLMETHYL METHACRYLATE  
 $C_{17}H_{16}O_2$



H. KAGEYAMA, K. MIKI, N. TANAKA and N. KASAI, 1982. *Makromol. Chem.*, **183**, 2863-2870.

Monoclinic,  $C2/c$ ,  $a = 29.177$ ,  $b = 6.017$ ,  $c = 16.110$  Å,  $\beta = 96.840^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.071$  for 1768 reflexions.

Of the four possible structures of methyl methacrylate derivatives, the present molecule has an antiperiplanar-synperiplanar (ap,sp) structure (Fig. 1). The molecule consists of three planar portions, the methacryl residue and two phenyl

groups. The methacryl residue is comparatively poorly planar and can be divided into two highly planar portions (C(1),C(2),C(3),C(4) and O(1),O(2),C(1),C(2) whose dihedral angle is  $8.3^\circ$ ). The dihedral angles between the methacryl residue plane and the phenyl ring planes are  $36.4$  and  $74.0^\circ$  respectively for the C(6) and C(12) phenyl rings. Bond distances of interest are: O(1)-C(1)  $1.340$ , O(2)-C(1)  $1.194$ , C(2)-C(3)  $1.332$ , C(5)-C(6)  $1.507$ , O(1)-C(5)  $1.456$ , C(1)-C(2)  $1.492$ , C(2)-C(4)  $1.466$  and C(5)-C(12)  $1.512$  Å.

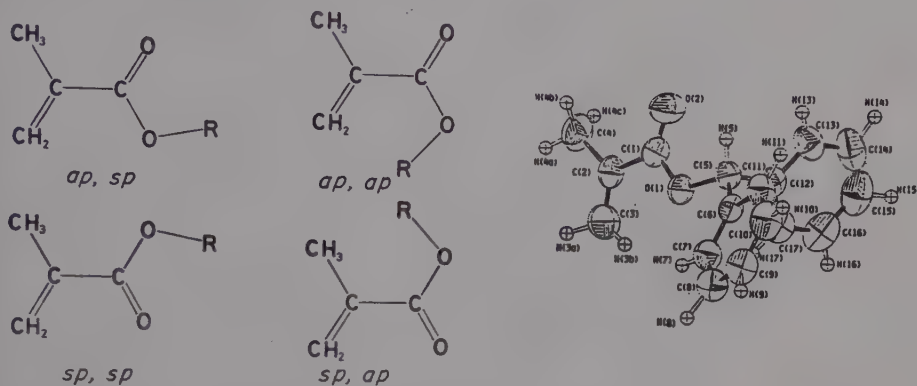


Fig. 1. The four possible structures of methacrylates (ap = antiperiplanar, sp = synperiplanar) (left), and the molecular structure of  $C_{17}H_{16}O_2$  (right).

#### METHYL 1-METHOXY-2-PHENYLTHIO CINNAMATE

$C_{17}H_{16}O_2S$

S. SINGH, M.M. BHADHBADE, K. VENKATESAN and V. RAMAMURTHY, 1982. J. Org. Chem., 47, 3550-3553.

Monoclinic,  $P2_1/n$ ,  $a = 8.729$ ,  $b = 20.227$ ,  $c = 9.523$  Å,  $\beta = 14.69^\circ$ ,  $D_m = 1.24$ ,  $Z = 4$ . Cu radiation,  $R = 0.070$  for 2002 reflexions.

The analysis shows that the molecule has cis geometry (cis-cinnamate) (Fig. 1) and is s-transoid with respect to the  $\alpha,\beta$ -unsaturated thiocarbonyl chromophore.

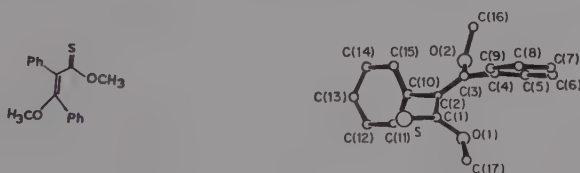


Fig. 1. The  $C_{17}H_{16}O_2S$  molecule.

#### POTASSIUM HYDROGEN DIFORMATE

$C_2H_3KO_4$  (I)

#### RUBIDIUM HYDROGEN DIFORMATE

$C_2H_3O_4Rb$  (II)

#### CESIUM HYDROGEN DIFORMATE

$C_2H_3CsO_4$  (III)

V.K. TRUNOV, Yu.A. VELIKODNI, A.D. CHUBINIDZE and T.M. SAS, 1982. Koord. Khim., 8, 94-97.

I. Orthorhombic,  $Pbca$ ,  $a = 7.374$ ,  $b = 17.703$ ,  $c = 7.504$  Å,  $Z = 8$ . Mo radiation,  $R = 0.035$  for 1061 reflexions.

II. Orthorhombic,  $Pbca$ ,  $a = 7.618$ ,  $b = 17.940$ ,  $c = 7.867$  Å,  $Z = 8$ . (Isomorphous with I.)

III. Monoclinic,  $C2/c$ ,  $a = 7.653$ ,  $b = 17.915$ ,  $c = 7.852$  Å,  $\beta = 112.33^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.051$  for 1439 reflexions.

The crystal structure of III along with bond lengths is shown in Fig. 1. In I the mean K-O distance is 2.887 Å.

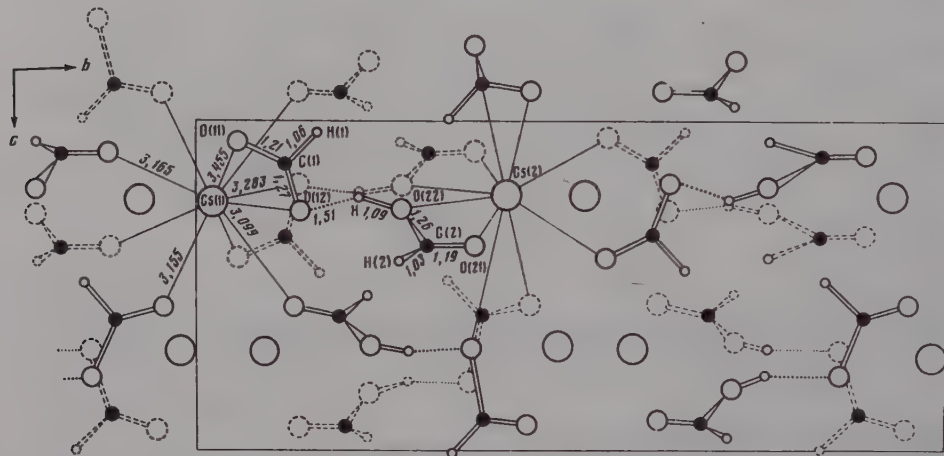


Fig. 1.  $C_2H_3CsO_4$ : the crystal structure showing bond lengths.

#### SODIUM HYDROGEN DIFORMATE



A.D. CHUBINIDZE, Ju.A. VELIKODNI and V.K. TRUNOV, 1982. Koord. Khim., 8, 855-858.

Triclinic,  $P\bar{1}$ ,  $a = 9.040$ ,  $b = 9.266$ ,  $c = 9.593$  Å,  $\alpha = 83.42^\circ$ ,  $\beta = 111.68^\circ$ ,  $\gamma = 109.91^\circ$ ,  $Z = 6$ . Mo radiation,  $R = 0.036$  for 1600 reflexions.

There are three molecules of sodium hydrogen diformate in the asymmetric unit. Each Na atom is surrounded by six O atoms with mean Na-O distances of 2.421, 2.406 and 2.415 Å. The C(1)-O(11) and C(1)-O(12) distances (Fig. 1) are 1.235(3) and 1.250(3) Å respectively, while C(2)-O(21) and C(2)-O(22) are 1.201(4) and 1.289(4) Å respectively and C(3)-O(31) and C(3)-O(32) are 1.201(3) and 1.286(3) Å respectively. There is extensive hydrogen bonding in the crystal.



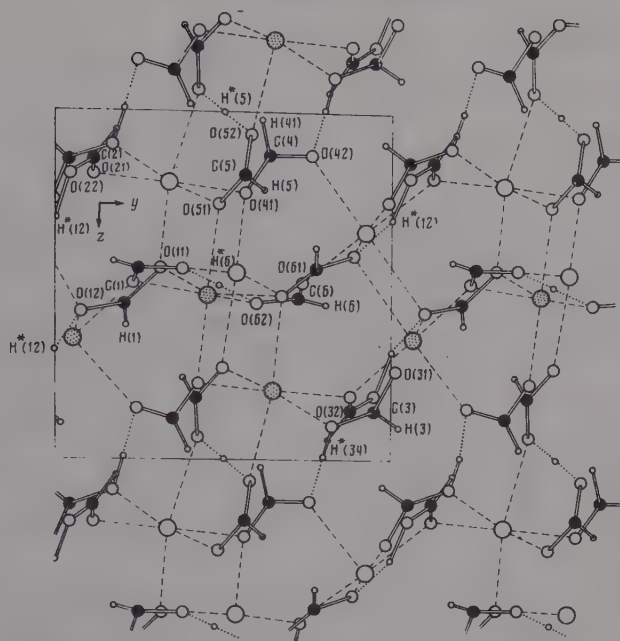
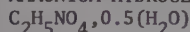


Fig. 1.  $C_2H_3NaO_4$ : (100) projection of the crystal structure.

# AMMONIUM HYDROGEN OXALATE HEMIHYDRATE



H.L. KELLER, D. KUCHARCZYK and H. KUPPERS, 1982. *Z. Krist.*, **158**, 221-232.

Monoclinic,  $P2_1/n$ ,  $a = 12.331$ ,  $b = 11.2241$ ,  $c = 6.7608$  Å,  $\beta = 91.64^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.075$  for 2738 reflexions (at 128 K).

For details of the orthorhombic room-temperature phase see (1). The structural change on cooling to 128 K is mainly characterized by the  $NH_4^+$  ions and water molecules moving off their special positions on mirror planes in the orthorhombic system. This leads to rearrangements within the hydrogen bond network. Some dimensions are in Fig. 1.

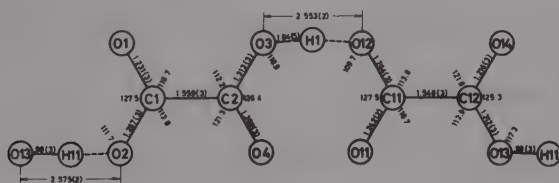
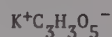
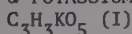


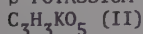
Fig. 1. Dimensions in the low-temperature form of  $C_2H_5NO_4 \cdot 0.5(H_2O)$ .

## 1. Structure Reports, **39B**, 12.

### $\alpha$ -POTASSIUM HYDROGEN TARTRONATE



### $\beta$ -POTASSIUM HYDROGEN TARTRONATE



J. KROON and J.A. KANTERS, 1982. *Croat. Chem. Acta*, **55**, 99-104.

I. Monoclinic,  $P2_1/n$ ,  $a = 7.356$ ,  $b = 8.116$ ,  $c = 9.197$  Å,  $\beta = 94.81^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.047$  for 1129 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 6.532$ ,  $b = 9.248$ ,  $c = 9.505$  Å,  $\beta = 99.74^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.047$  for 1204 reflexions.

The most striking difference between the dimorphs is the hydrogen bonding scheme. The  $\alpha$ -form has A-type bonds and the  $\beta$ -form B-type hydrogen bonds (A-type bonds connect carboxyl groups related by translational-free symmetry, B-type bonds connect carboxyl groups which are crystallographically-independent). In I, selected distances are:  $O(2)\dots O(2')$  2.433,  $O(4)\dots O(4')$  2.436 Å and in II,  $O(2)\dots O(5''')$  2.484,  $O(6)\dots O(4''''')$  2.859 Å. The  $K^+$  ions are eightfold coordinated by O atoms, the mean  $K\cdots O$  distances in I and II being 2.89 and 2.86 Å respectively (Fig. 1).

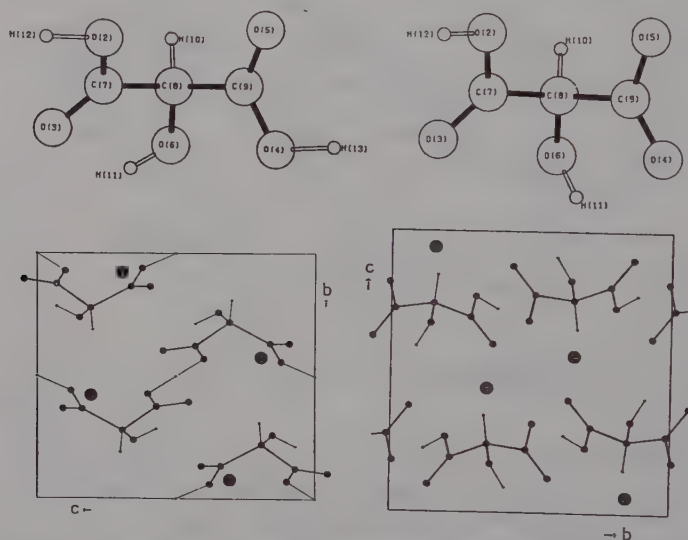


Fig. 1. Atomic numbering in the anions ( $\alpha$ -form top left,  $\beta$ -form top right), and projections of crystal structures down  $[100]$  of the  $\alpha$  form (bottom left) and  $\beta$  form (bottom right) of  $C_3H_3KO_5$ .

POTASSIUM TRIHYDROGEN DIOXALATE DIHYDRATE (POTASSIUM TETROXALATE)  
 $C_3H_3KO_8 \cdot 2H_2O$

$KH_3(C_2O_4)_2 \cdot 2H_2O$

C.J. GILMORE and J.C. SPEAKMAN, 1982. *Acta Cryst.*, **B38**, 2809-2813.

Triclinic,  $P\bar{1}$ ,  $a = 7.027$ ,  $b = 10.595$ ,  $c = 6.356$  Å,  $\alpha = 101.40$ ,  $\beta = 100.12$ ,  $\gamma = 93.80^\circ$ ,  $D_m = 1.86$ ,  $Z = 2$ . Mo radiation,  $R = 0.030$  for 2422 reflexions.

The crystal structure of potassium tetroxalate (Fig. 1) has previously been determined in  $P\bar{1}$  (1) and  $P1$  (2) and the present work was undertaken to confirm  $P\bar{1}$  as the correct space group. The results are in very good agreement with the initial earlier work (1). In the crystal structure  $K\cdots O$  distances are 2.867 to 2.921 Å and  $O\cdots O$  distances in the extensive network of hydrogen bonds are 2.480 to 2.835 Å.

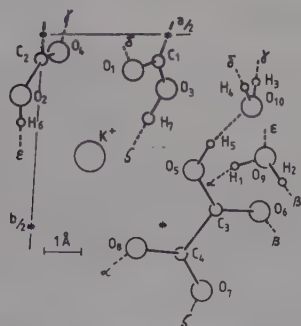
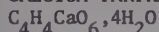


Fig. 1.  $\text{KH}_3(\text{C}_2\text{O}_4)_2 \cdot 2\text{H}_2\text{O}$ : the asymmetric unit projected normal to (001); hydrogen bonds are indicated by broken lines.

1. Structure Reports, 29, 465.

2. Ibid, 48B, 23.

#### CALCIUM TARTRATE TETRAHYDRATE



F.C. HAWTHORNE, I. BORYS and R.B. FERGUSON, 1982. Acta Cryst., B38, 2461-2463.

Orthorhombic,  $\text{P2}_1\text{2}_1\text{2}_1$ ,  $a = 9.631$ ,  $b = 10.573$ ,  $c = 9.215 \text{ \AA}$ ,  $Z = 4$ . Mo radiation,  $R = 0.037$  for 1504 reflexions.

In the crystal structure calcium is surrounded by eight oxygens at distances between 2.377(3) and 2.518(3) Å, arranged in a distorted Siamese dodecahedron (Fig. 1). Calcium links the tartrate molecules into infinite chains parallel to  $c$ , with one  $\text{Ca} \cdots \text{O}$  contact linking adjacent chains in the  $a$  direction. Most interchain linkage is provided by the network of hydrogen bonds formed by the additional water molecules. Bond lengths and angles in the tartrate molecule are normal.

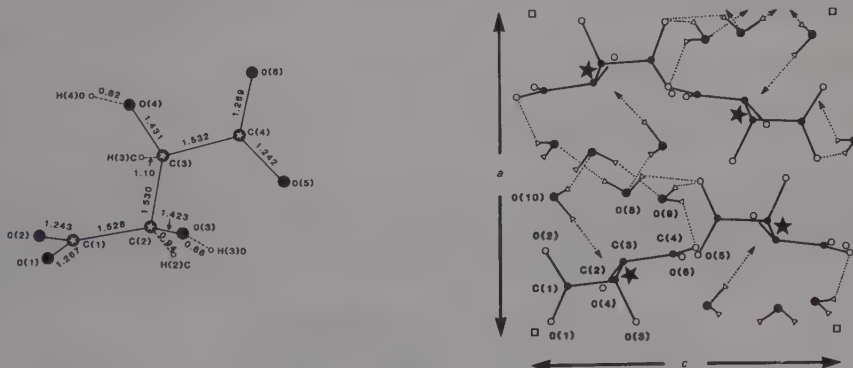
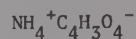


Fig. 1.  $\text{Ca}^{2+}(\text{O}_2\text{C}, \text{CHOH}, \text{CHOH}, \text{CO}_2)^{2-} \cdot 4\text{H}_2\text{O}$ : bond lengths in the tartrate ion and a projection of the crystal structure down the  $b$  axis; Ca is represented by stars.

#### AMMONIUM HYDROGEN MALEATE



L. GOLIČ and I. LEBAN, 1982. Croat. Chem. Acta, 55, 41-45.

Orthorhombic,  $\text{Pbcm}$ ,  $a = 4.616$ ,  $b = 8.085$ ,  $c = 16.410 \text{ \AA}$ ,  $D_m = 1.46$ ,  $Z = 4$ . Mo

radiation,  $R = 0.052$  for 690 reflexions.

The crystal structure is essentially the same as that of potassium hydrogen maleate (1). It is composed of alternate layers of ammonium and planar hydrogen maleate ions (Fig. 1). The cation has crystallographic twofold symmetry whilst the anion has crystallographic  $m$  symmetry with the acidic H atom lying on the mirror plane. Distances within the anion are:  $C(1)-O(1)$  1.277,  $C(1)-O(2)$  1.243,  $C(1)-C(2)$  1.490,  $C(2)-C(2')$  1.329 and  $C(2)-HC$  0.96 Å. The  $O(1)...O(1')$  distance is 2.432 Å and the  $O(1)-H-O(1')$  angle  $174^\circ$ . Atom H(1) is involved in a hydrogen bond  $N-H(1)...O(1)$  (3.146 Å) with an angle of  $166^\circ$  whilst H(2) forms a bifurcated hydrogen bond to  $O(2')$  and  $O(2'')$  with  $N...O$  distances of 2.930 and 3.003 Å respectively.

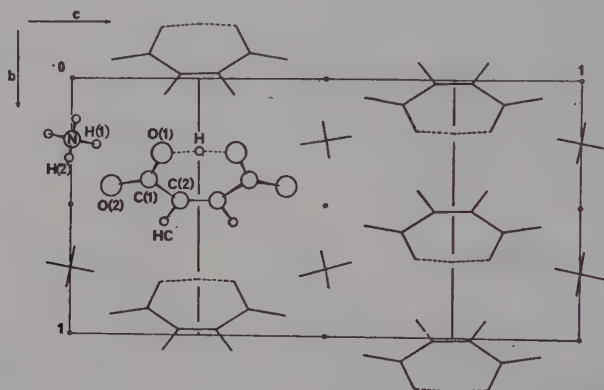


Fig. 1. View of the crystal structure of  $C_4H_7NO_4$  along  $[100]$  direction.

1. Structure Reports, 26, 555.

AMMONIUM HYDROGEN OXYDIACETATE  
 $C_4H_9NO_5$



H. HERBERTSSON and B. HEDMAN, 1982. Acta Cryst., B38, 320-322.

Monoclinic,  $I2/c$ ,  $a = 10.1298$ ,  $b = 17.9857$ ,  $c = 7.0795$  Å,  $\beta = 91.286^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.041$  for 996 reflexions.

The crystal structure (Fig. 1) is composed of chains of oxydiacetate ions along  $b$ . The oxydiacetate residues are linked end-to-end by short hydrogen bonds ( $O...O$  2.450 Å) into infinite chains forming layers parallel to the  $ab$  plane. The layers are held together by  $NH_4^+$  ions which are coordinated tetrahedrally by four O atoms with  $N...O$  distances 2.891-3.012 Å. The shortest  $N...N$  distance is 3.558 Å.

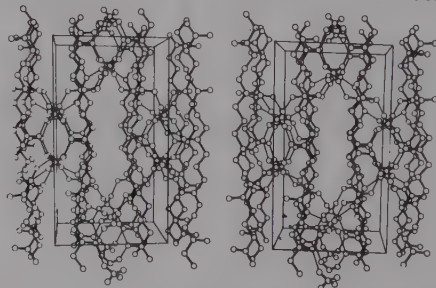


Fig. 1. A stereoscopic view of the crystal structure of ammonium hydrogen oxydiacetate; the  $O-H...O$  bonds are drawn unfilled, and the  $H...O$  bonds from  $NH_4^+$  are drawn with thin lines.

## THALLIUM TRIACETATE MONOHYDRATE



R. FAGGIANI and I.D. BROWN, 1982. *Acta Cryst.*, **B38**, 2473-2475.

Monoclinic,  $P2_1/c$ ,  $a = 9.311$ ,  $b = 14.341$ ,  $c = 9.198 \text{ \AA}$ ,  $\beta = 119.69^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.038$  for 577 reflexions.

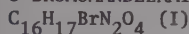
The Tl atom is irregularly coordinated by eight O atoms ( $\text{Tl} \cdots \text{O}$  2.17-2.78 Å) (Fig. 1). The angles between adjacent bonds correlate with the strengths of the bonds. The molecules are linked by a bridging acetate O atom and a hydrogen bond into columns running along  $c$ . There is a simple relation between the structure of the monohydrate and the anhydrous triacetate (1).



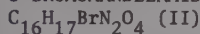
Fig. 1.  $\text{Tl}(\text{CH}_3\text{CO}_2)_3 \cdot \text{H}_2\text{O}$ : stereoscopic view.

1. *Structure Reports*, **44B**, 661.

## o-BROMOMANDELAMIDINIUM MANDELATE R-(-).R-(-) (threo) ISOMER



## o-BROMOMANDELAMIDINIUM MANDELATE S-(+).R-(-) (erythro) ISOMER



J. IBALL, J.N. LOW, D.G. NEILSON and C.H. MORGAN, 1982. *Cryst. Struct. Comm.*, **11**, 349-354.

I. Orthorhombic,  $P2_12_12_1$ ,  $a = 5.84$ ,  $b = 10.26$ ,  $c = 27.63 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.095$  for 1346 reflexions.

II. Monoclinic,  $P2_1$ ,  $a = 11.91$ ,  $b = 6.14$ ,  $c = 11.39 \text{ \AA}$ ,  $\beta = 99.6^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.086$  for 1310 reflexions.

A view of each structure is shown in Fig. 1. In each case the ions are linked by hydrogen bonding. The more stable isomer (II) has a puckered ring structure allowing for maximum neutralisation of the formal charges on the amidinium and carboxylate ions.

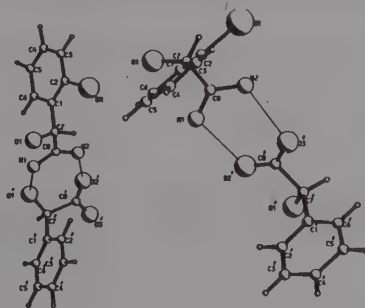
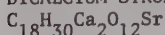


Fig. 1. The two mandelate derivatives, (I) (left) and (II) (right).



## DICALCIUM STRONTIUM PROPIONATE



N. MISHIMA, K. ITOH and E. NAKAMURA, 1982. J. Phys. Soc. Japan, 51, 3958-3965.

Tetragonal,  $P4_1$ ,  $a = 12.475$ ,  $c = 17.141$  Å,  $Z = [4]$ . Cu radiation,  $R = 0.044$  for 2312 reflexions (at  $-40^\circ\text{C}$ ).

This study has revealed that four  $\beta$ -carbon atoms C(13), C(23), C(3a3) and C(3b3) which are in a perfectly disordered state in the paraelectric phase (1), are in a partially disordered state at  $-40^\circ\text{C}$  (Fig. 1). The Sr-O distances range from 2.608 to 3.835 Å, and the Ca-O distances from 2.254 to 2.366 Å at  $-40^\circ\text{C}$  compared to 2.611 to 3.705 Å and 2.268 to 2.368 Å for Sr-O and Ca-O respectively in the paraelectric phase. See also (2)

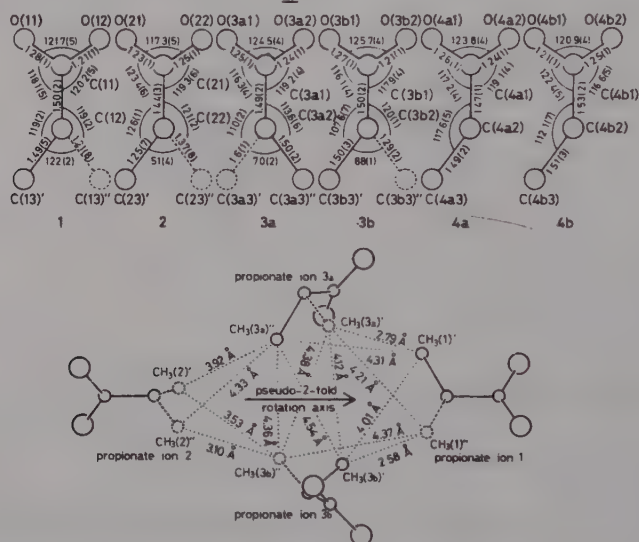
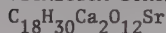


Fig. 1. Bond lengths and angles in the six independent propionate ions in  $\text{C}_{18}\text{H}_{30}\text{Ca}_2\text{O}_{12}\text{Sr}$  (top), and interatomic distances between the methyl groups of the propionate ions 1, 2, 3a and 3b viewed along the  $c$  axis.

1. Structure Reports, 48B, 27.
2. Following Report.

## DICALCIUM STRONTIUM PROPIONATE (Ferroelectric Phase)



K. STADNICKA, A.M. GLAZER, S. SINGH and J. SLIWINSKI, 1982. J. Phys. C:Solid State Phys., 15, 2577-2586.

Tetragonal,  $P4_3$ ,  $a = 12.452$ ,  $c = 17.217$  Å,  $Z = 4$ . Cu radiation,  $R = 0.072$  for 2811 reflexions (at  $-30^\circ\text{C}$ ).

There are six independent propionate groups in the structure, two of which have statistical disorder of their methyl carbon atoms between two sites. The average Sr-O and Ca-O dimensions are 2.73(7) and 2.31(5) Å respectively. For related work see (1) and (2).

1. Structure Reports, 23, 536.
2. Preceding Report.

## PROPIONAMIDE



A. USANMAZ and G. ADLER, 1982. *Acta Cryst.*, **B38**, 660-662.

Monoclinic,  $P2_1/c$ ,  $a = 8.728$ ,  $b = 5.734$ ,  $c = 9.790$  Å,  $\beta = 115.74^\circ$ ,  $D_m$  (293 K) = 1.046,  $Z = 4$ . Cu radiation,  $R = 0.084$  for 394 reflexions (at 123 K).

Excluding H atoms, the propionamide molecules are planar and form dimers (Fig. 1). Some dimensions are, C-O 1.254(9), C-N 1.326(10), C(1)-C(2) 1.476(18), C(2)-C(3) 1.502(6) Å, C(1)-C(2)-C(3)  $115.6^\circ$ .

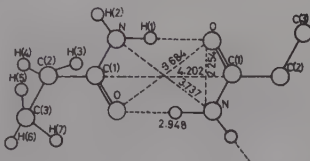


Fig. 1. The propionamide dimer with some distances in Å.

## 1,3-DIAMINOPROPANE DIHYDROCHLORIDE



J. BRISSON and F. BRISSE, 1982. *J. Cryst. Spectrosc. Res.*, **12**, 39-43.

Orthorhombic,  $Pbcn$ ,  $a = 8.567$ ,  $b = 9.341$ ,  $c = 9.444$  Å,  $D_m = 1.30$ ,  $Z = 4$ . Cu radiation,  $R = 0.034$  for 781 reflexions.

A refinement with new data of a structure determined previously (1). The cation lies on a twofold axis and dimensions are in Fig. 1 along with details of hydrogen bonding N-H...Cl.

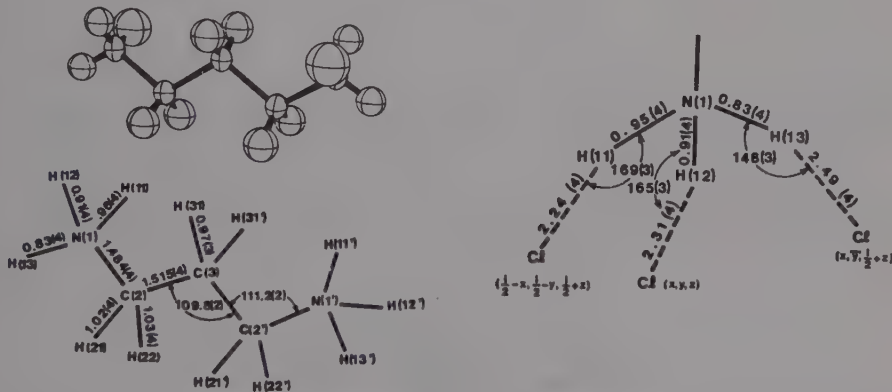
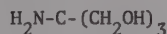


Fig. 1. The trimethylenediammonium cation with dimensions and hydrogen bonds.

1. S. HIROKAWA, M. MASAKUNI, M. SEKO and T. NOGUCHI, 1968. *Mem. Def. Acad. Jpn.*, **8**, 485-498.

## TRIS (HYDROXYMETHYL) AMINOMETHANE

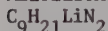


E. KENDI, 1982. *Z. Krist.*, **160**, 139-143.

Orthorhombic,  $Pna2_1$ ,  $a = 8.807$ ,  $b = 8.872$ ,  $c = 7.709$  Å,  $D_m = 1.335$ ,  $Z = 4$ . Cu radiation,  $R = 0.080$ .

The central C atom has distorted tetrahedral geometry, with N-C-C 100.9-108.8(14)° and C-C-C 103.9-120.2(24)°, N-C 1.63(3), C-C 1.45(4)-1.54(2), C-O 1.41-1.51(2) Å.

ALLYLLITHIUM N,N,N',N'-TETRAMETHYLETHYLENEDIAMINE



H. KOSTER and E. WEISS, 1982. Chem. Ber., 115, 3422-3426.

Orthorhombic, Cmc2<sub>1</sub>, a = 13.424, b = 7.998, c = 11.024 Å, Z = 8. R = 0.102 for 285 reflexions.

Each Li atom is coordinated (Fig. 1) to a terminal C atom from two different allyl groups thus producing polymeric chains. The tetrahedral coordination of Li is completed by the two N atoms of an ethylenediamine (Li-C 2.21(4), 2.30(4); Li-N 2.19(2) Å). The monomeric units have C<sub>s</sub> symmetry.

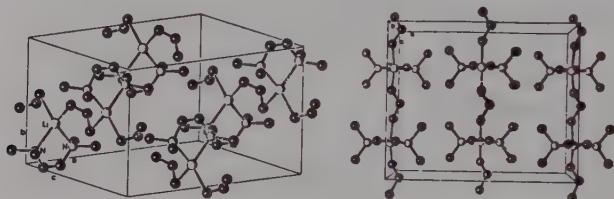


Fig. 1. Two views of the unit cell contents of  $\text{C}_6\text{H}_{16}\text{N}_2, \text{Li}^+, \text{C}_3\text{H}_5^-$ .

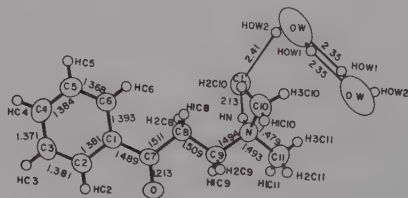
3-DIMETHYLAMINO-1-PHENYL-1-PROPANONE HYDROCHLORIDE MONOHYDRATE



E.O. SCHLEMPER, T.S. MANSOUR and E.M. KAISER, 1982. Acta Cryst., B38, 625-627.

Orthorhombic, Pbca, a = 7.342, b = 11.043, c = 30.440 Å, Z = 8. Mo radiation, R = 0.044 for 1477 reflexions.

The structure contains quaternary ammonium cations and chloride anions linked by N-H...Cl hydrogen bonds. The water molecule is weakly hydrogen bonded to a symmetry related one and to the Cl<sup>-</sup> ion. Except for C(10) (Fig. 1) the non-hydrogen atoms in the cation are coplanar within 0.27 Å.



S(+) 2-ETHYLAMINO-1-(3-TRIFLUOROMETHYLPHENYL)PROPANE HYDROCHLORIDE (FENFLURAMINE HYDROCHLORIDE)

$C_{12}H_{17}ClF_3N$

M. MAYER, G. PEREZ, M.N. PETIT and G. COQUEREL, 1982. Cryst. Struct. Comm., 11, 1853-1856.

Monoclinic,  $P2_1$ ,  $a = 11.885$ ,  $b = 7.134$ ,  $c = 8.304 \text{ \AA}$ ,  $\beta = 97.50^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.073$  for 2318 reflexions.

Bond lengths in the fenfluramine cation are shown in Fig. 1.

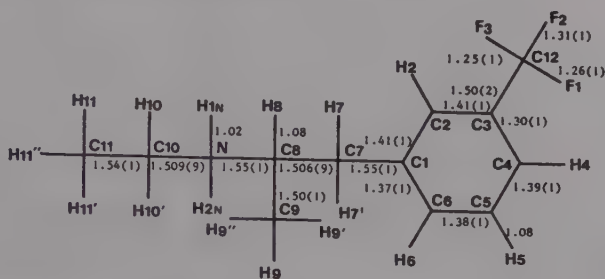


Fig. 1. Bond lengths in S(+) fenfluramine hydrochloride.

t-BUTYL[ETHYL-2-(3,5-DIHYDROXYPHENYL)-2-HYDROXY]AMMONIUM SULPHATE HYDRATE (TERBUTALINE SULPHATE HYDRATE)

$C_{12}H_{20}NO_5S \cdot 0.5, 1.5(H_2O)$

$[NH_2(C_4H_9)(C_8H_9O_3)]^+, 0.5SO_4^{2-}, 1.5H_2O$

D. HICKEL, A. CARPY, M. LAGUERRE and J.-M. LEGER, 1982. Acta Cryst., B38, 632-635.

Triclinic,  $P\bar{1}$ ,  $a = 10.984$ ,  $b = 11.935$ ,  $c = 14.521 \text{ \AA}$ ,  $\alpha = 62.49^\circ$ ,  $\beta = 62.69^\circ$ ,  $\gamma = 78.80^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.076$  for 5742 reflexions.

In one of the two independent molecules, atom O(8) (Fig. 1) is disordered over two positions; otherwise the molecules are very similar. The two C(3)-C(4)-C(7)-C(9) torsion angles are  $59$  and  $62^\circ$  and the C(4)-C(7)-C(9)-N(10) angles are  $180$  and  $182^\circ$ . There is extensive N-H...O and O-H...O hydrogen bonding linking cations, anions and water molecules. The S-O bond lengths average  $1.457 \text{ \AA}$ .

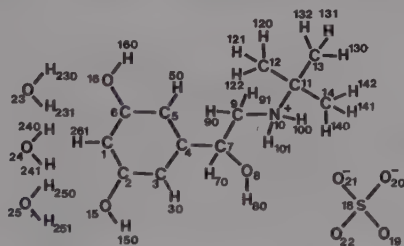


Fig. 1. Atom numbering in terbutaline sulphate hydrate.

PROCAINE

$C_{13}H_{20}N_2O_2$

S. KASHINO, M. IKEDA and M. HAISA, 1982. Acta Cryst., B38, 1868-1870.

Monoclinic,  $P2_1/a$ ,  $a = 11.56$ ,  $b = 14.38$ ,  $c = 8.202 \text{ \AA}$ ,  $\beta = 98.0^\circ$ ,  $D_m = 1.17$ ,  $Z = 4$ . Cu radiation,  $R = 0.098$  for 1690 reflexions.

Within the molecule the benzene ring has the quinoid structure (C(2)-C(3) 1.369(6), C(5)-C(6) 1.360(6)  $\text{\AA}$ ) and the angle between the p-aminophenyl plane and the C(1),C(7),O(8),O(9),C(11) plane is  $1.6(2)^\circ$ . The conformation of the C-O-C-C-N side chain is trans-gauche ( $\tau(\text{C-O-C-C})$   $147.1(4)$ ,  $\tau(\text{O-C-C-N})$   $-82.6(5)^\circ$ ). Molecules are hydrogen bonded into double-layered sheets on (001) through the carbonyl O accepting two bonds from amino groups (Fig. 1).

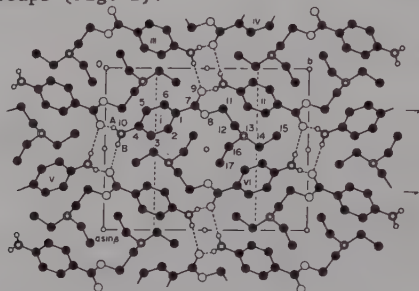


Fig. 1. The crystal structure of procaine projected along  $c$ . (Broken lines show hydrogen bonds).

N-ISOPROPYL-N-METHYL-DI-*t*-BUTYLKETENIMINIUM HEXACHLOROANTIMONY  
 $\text{C}_{14}\text{H}_{28}\text{Cl}_6\text{NSb}$

$\text{C}_{14}\text{H}_{28}\text{N}^+, \text{SbCl}_6^-$

J. LAMBRECHT, L. ZSOLNAI, G. HUTTNER and J.C. JOCHIMS, 1982. Chem. Ber., 115, 172-184.

Monoclinic,  $P2_1/c$ ,  $a = 7.98$ ,  $b = 22.51$ ,  $c = 12.88 \text{ \AA}$ ,  $\beta = 97.8^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.041$  for 2140 reflexions (at 233 K).

The cation (Fig. 1) has approximate  $C_{2v}$  symmetry with C(2)-C(1)-N(1)  $176(1)^\circ$ . The two *t*-butyl and isopropyl substituents adopt conformations such that a methyl of each (C(43),C(33),C(51)) is nearly eclipsed with respect to the double bond.

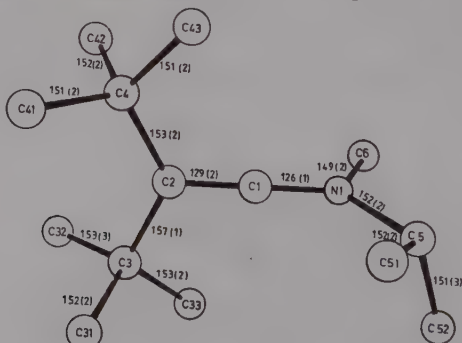


Fig. 1. The  $\text{C}_{14}\text{H}_{28}\text{N}^+$  cation with bond lengths (pm) and e.s.d.'s.

1-ISOPROPYLAMINO-3-(1-NAPHTHYLAMINO)-PROPAN-2-OL (AZAPROPRANOLOL)  
 $\text{C}_{16}\text{H}_{22}\text{N}_2\text{O}$

M. LAGUERRE, J.-M. LÉGER, D. MERLET, J.-C. COLLETER and J.-P. DUBOST, 1982. Acta Cryst., B38, 2291-2293.

Monoclinic,  $P2_1/c$ ,  $a = 14.386$ ,  $b = 11.128$ ,  $c = 9.796$  Å,  $\beta = 108.28^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.075$  for 2526 reflexions.

The structure of the title compound has been compared with that of propranolol hydrochloride (I) (Fig. 1). The value of the torsion angle  $C(1)-N(12)-C(13)-C(14)$ ,  $285.6^\circ$ , differs greatly from the corresponding angle in propranolol,  $180.4^\circ$ , where  $N(12)$  is replaced by O. As a result, the distance from the N(isopropylamino) atom to the least-squares plane of the aromatic rings is much longer in the title compound (3.1 Å) than in propranolol (1.2 Å).

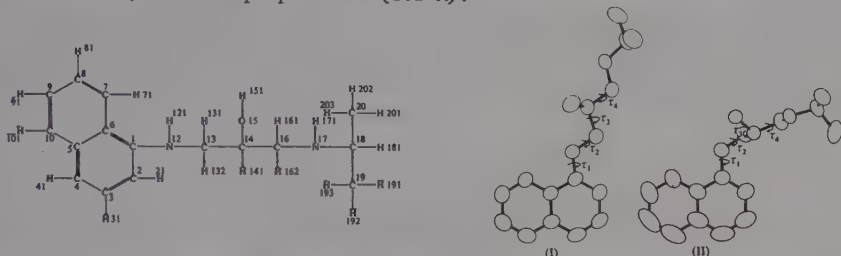


Fig. 1. Azapropranolol and propranolol: molecular structure of azapropranolol (left) and perspective views of propranolol (I) and azapropranolol (II) (right).

# 1. Structure Reports, 41B, 188.

4-[ (4-CHLOROPHENYL) (5-FLUORO-2-HYDROXYPHENYL)METHYLENEAMINO]BUTANAMIDE (PROGABIDE)  
 $C_{17}H_{16}ClFN_2O_2$

C. PASCARD, J.-P. KAPLAN, B. RAIZON and B. MOMPON, 1982. Acta Cryst., B38, 3131-3132.

Monoclinic,  $Cc$ ,  $a = 11.018$ ,  $b = 15.295$ ,  $c = 9.736$  Å,  $\beta = 97.63^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.064$  for 1107 reflexions.

A six-membered ring (Fig. 1) is formed by means of the hydrogen bond between  $O(1)$  and  $N(1)$ . This ring is nearly coplanar with that of ring II. Rings I and II are perpendicular ( $89.5^\circ$ ). The amido plane makes an angle of  $123^\circ$  with the chlorinated benzene ring (the shortest distance between these two groups is  $N(2) \dots C(7)$ :  $3.94(2)$  Å). The molecules are held together through hydrogen bonds and van der Waals interactions.

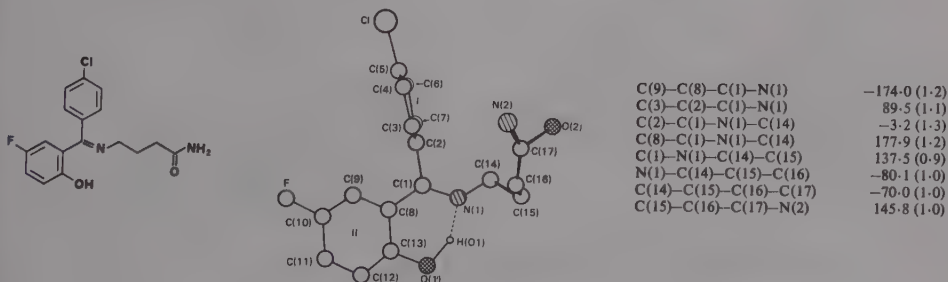


Fig. 1. A view of progabide and some torsion angles.



## (S)-N-BENZYL-1-METHYL-3-PHENYLPROPYLAMINE HYDROCHLORIDE

 $C_{17}H_{22}ClN$ 

P. MURRAY-RUST, J. MURRAY-RUST, D. HARTLEY and J. CLIFTON, 1982. Acta Cryst., B38, 306-308.

Monoclinic,  $P2_1$ ,  $a = 5.56$ ,  $b = 9.97$ ,  $c = 14.41$  Å,  $\beta = 80.42^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.044$  for 2289 reflexions.

The crystal structure consists of cations (Fig. 1) and chloride anions held together by two weak hydrogen bonds (N-H...Cl) of 3.15 Å. The backbone of the molecule is almost fully extended and the maximum deviation from perfect staggering is  $17^\circ$ . The phenyl ring [C(41)] is nearly perpendicular to the chain. The absolute configuration is conformed as S.

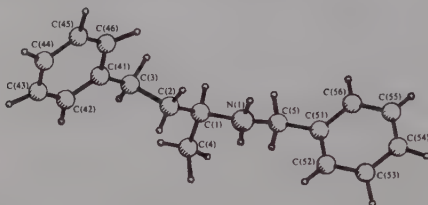


Fig. 1. A perspective view of the  $C_{17}H_{22}N^+$  cation.

## (-)-erythro-5-METHYLMETHADONE PERCHLORATE

 $C_{22}H_{31}ClNO_5$  $C_{22}H_{31}NO^+, ClO_4^-$ 

P.S. PORTOGHESE, J.H. POUPAERT, D.L. LARSON, W.C. GROUTAS, G.D. MEITZNER, D.C. SWENSON, G.D. SMITH and W.L. DUAX, 1982. J. Med. Chem., 25, 684-688.

Orthorhombic,  $P2_12_12_1$ ,  $a = 10.823$ ,  $b = 23.56$ ,  $c = 8.735$  Å,  $Z = 4$ . Mo radiation,  $R = 0.068$  for 1360 reflexions.

The absolute configuration of the title compound has been established by X-ray crystallographic analysis of its perchlorate salt. This enantiomer was found to have 5S,6S chirality as defined by the seven torsion angles in Fig. 1. R and R' are both  $CH_3$ , and the definitions and values of  $\tau_1$ ,  $\tau_2$ ,  $\tau_3$ ,  $\tau_4$ ,  $\tau_5$ ,  $\tau_6$  and  $\tau_7$  are respectively C(11)-C(10)-C(4)-C(5)  $-40^\circ$ , C(17)-C(16)-C(4)-C(5)  $100^\circ$ , C(3)-C(4)-C(5)-C(6)  $64^\circ$ , C(1)-C(2)-C(3)-O(3)  $-10^\circ$ , O(3)-C(3)-C(4)-C(5)  $0^\circ$ , C(4)-C(5)-C(6)-H  $97^\circ$ , C(5)-C(6)-N-C(8)  $92^\circ$ , C(5)-C(6)-N-C(9)  $-156^\circ$ .

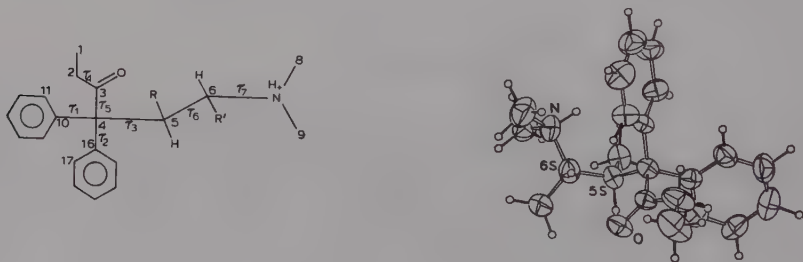
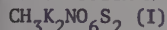
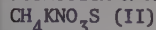


Fig. 1. Torsion angles in, and molecular structure of  $C_{22}H_{31}NO^+$ .

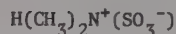
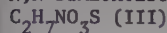
## DIPOTASSIUM N-METHYLNITROGENBIS(TRIOXOSULPHATE)



## POTASSIUM N-METHYLIMIDO(TRIOXOSULPHATE)



## N,N-DIMETHYLSULPHAMIC ACID



A.J. MORRIS, C.H.L. KENNARD, J.R. HALL and G. SMITH, 1982. *Inorg. Chim. Acta*, **62**, 247-252.

I. Monoclinic,  $P2_1/c$ ,  $a = 9.819$ ,  $b = 6.053$ ,  $c = 14.749$  Å,  $\beta = 113.17^\circ$ ,  $D_m = 2.18$ ,  $Z = 4$ . Mo radiation,  $R = 0.075$  for 376 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 9.468$ ,  $b = 7.256$ ,  $c = 7.306$  Å,  $\beta = 95.01^\circ$ ,  $D_m = 1.99$ ,  $Z = 4$ . Mo radiation,  $R = 0.039$  for 439 reflexions.

III. Monoclinic,  $P2_1/c$ ,  $a = 7.704$ ,  $b = 6.136$ ,  $c = 11.900$  Å,  $\beta = 101.76^\circ$ ,  $D_m = 1.49$ ,  $Z = 4$ . Mo radiation,  $R = 0.063$  for 438 reflexions.

In I a flattened pyramidal geometry is found for the N atom (Fig. 1) with angles S(1)-N-S(2)  $120^\circ$ , S(1)-N-C  $117^\circ$ , S(2)-N-C  $119^\circ$ . The N atom is 0.17 Å out of the plane through C, S(1) and S(2). Selected distances are: N-S(1) 1.70, N-S(2) 1.80, N-C 1.34 Å. In II and III, tetrahedral stereochemistry for nitrogen was observed. In II, S-N-C  $116.4^\circ$  and in III, C(1)-N-C(2)  $111.5^\circ$ , S-N-C(1)  $112.7^\circ$  and S-N-C(2)  $112.0^\circ$ . Important bond distances in II are: N-S 1.637, N-C 1.469 Å, and in III are: N-S 1.790, N-C (mean) 1.504 and S-O (mean) 1.430 Å. The N atom is out of the C,S,H(N) plane in II by 0.34 Å, and out of the plane C(1),C(2),S in III by 0.46 Å. In I the  $\text{K}^+$  cations are surrounded by eight O atoms from neighbouring molecules with K-O distances ranging from 2.72 to 3.13 Å. In II, one  $\text{K}^+$  is surrounded by eight O atoms and the second by six, in irregular arrangements with K-O distances ranging from 2.75 to 3.20 Å.

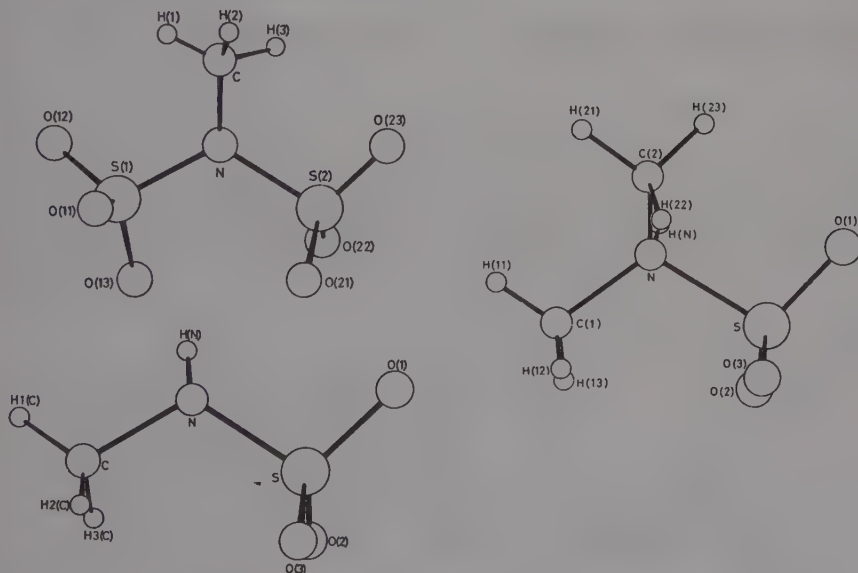


Fig. 1. The anions in I  $\text{CH}_3\text{K}_2\text{NO}_6\text{S}_2$  (top left), in II  $\text{CH}_4\text{KNO}_3\text{S}$  (bottom left) and the acid in III  $\text{C}_2\text{H}_7\text{NO}_3\text{S}$  (top right).

POTASSIUM N,N-DIMETHYLNITROGENTRIOXOSULPHATE HYDRATE  
 $C_2H_6KNO_3S \cdot 0.66(H_2O)$

$K^+[(CH_3)_2N(SO_3)]^-, 0.66(H_2O)$

A.J. MORRIS, C.H.L. KENNARD, J.R. HALL, G. SMITH and A.H. WHITE, 1982. *Inorg. Chim. Acta*, **65**, L153-L155.

Trigonal,  $P\bar{3}$ ,  $a = 10.96$ ,  $c = 11.88 \text{ \AA}$ ,  $Z = 6$ . Mo radiation,  $R = 0.149$  for 1110 reflexions.

In the anion the N atom has a distorted tetrahedral coordination and lies  $0.45 \text{ \AA}$  above the CCS plane (Fig. 1). Pertinent distances are: N-S  $1.684$ , N-C(1)  $1.47$ , N-C(2)  $1.51$ , S-O(1)  $1.403$ , S-O(2)  $1.465$ , S-O(3)  $1.428 \text{ \AA}$ . The anions are packed in layers in a head-to-head and tail-to-tail configuration in the  $ab$  plane. The water molecules are in the same plane and along with some of the  $K^+$  cations, lie in the spaces between layers.

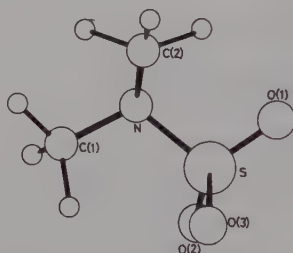


Fig. 1. The  $[(CH_3)_2N(SO_3)]^-$  anion viewed perpendicular to the CNS plane.

$\alpha, \alpha'$ -DITHIOBISFORMAMIDINIUM DINITRATE  
 $C_2H_8N_6O_6S_2$

$[(NH_2)_2C^+SSC(NH_2)_2][NO_3]_2$

G.B. JAMESON, E. BLAZSÖ, N. SEFERIADIS and H.R. OSWALD, 1982. *Acta Cryst.*, **B38**, 2272-2274.

Monoclinic,  $C2/c$ ,  $a = 10.871$ ,  $b = 8.113$ ,  $c = 11.580 \text{ \AA}$ ,  $\beta = 100.32^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.038$  for 1055 reflexions (at 110 K).

The dication (Fig. 1) has crystallographically imposed twofold symmetry with the following stereochemistry: C-N  $1.311(3)$  and  $1.301(3)$ , C-S  $1.786(2)$ , S-S  $2.022(1) \text{ \AA}$ ; planar configuration around the carbon atom; C-S-S-C torsional angle  $97.9(1)^\circ$ , S-S-C-N torsional angles  $6.6(2)$  and  $6.8(2)^\circ$ . An extensive hydrogen-bonding network links nitrate oxygen atoms with hydrogen atoms ( $O \cdots N$   $2.837(3)$ ,  $2.862(3)$ ,  $2.888(3)$  and  $2.970(3) \text{ \AA}$ ).

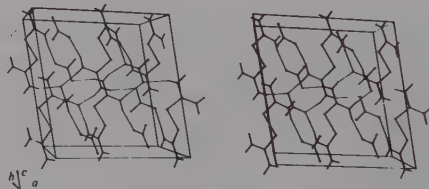
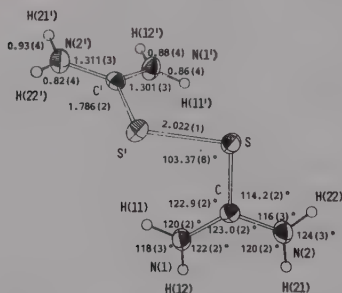
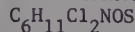


Fig. 1.  $[(NH_2)_2C^+SSC(NH_2)_2][NO_3]_2$ : bond distances and angles in the dication and a stereodiagram of the unit cell showing hydrogen bonds (thin lines).

## N-DICHLOROACETYL-S,S-DIETHYLSULPHILIMINE



A. KÁLMÁN, T. KORITSÁNSZKY, I. KAPOVITS and Á. KUCSMAN, 1982. Acta Cryst., B38, 1843-1845.

Monoclinic,  $P2_1/n$ ,  $a = 6.950$ ,  $b = 11.322$ ,  $c = 12.460$  Å,  $\beta = 95.32^\circ$ ,  $D_m = 1.468$ ,  $Z = 4$ . Mo radiation,  $R = 0.034$  for 2253 reflexions.

This redetermination after an earlier photographic analysis leads to an S-N bond length of 1.664(1) Å and to a short S...O contact of 2.838(1) Å giving an almost planar SNCO ring (Fig. 1).

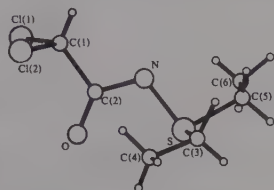
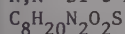


Fig. 1. The  $\text{C}_6\text{H}_{11}\text{Cl}_2\text{NOS}$  molecule.

## N,N'-DI-t-BUTYLSULPHAMIDE



J.L. ATWOOD, A.H. COWLEY, W.E. HUNTER and S.K. MEHROTRA, 1982. Inorg. Chem., 21, 435-437.

Monoclinic,  $P2_1/c$ ,  $a = 9.720$ ,  $b = 9.887$ ,  $c = 12.076$  Å,  $\beta = 90.46^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.030$  for 1242 reflexions.

The molecule is shown in Fig. 1. Both S-O distances are 1.434(2) Å, S-N 1.615(2) and 1.616(2) Å. The N atoms have nearly planar geometry, the S-N-C angles are expanded to 127.4(2) and 127.5(2)°.

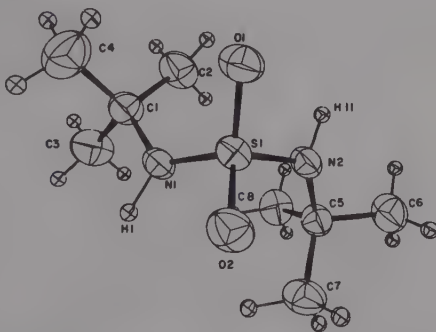
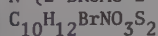


Fig. 1. The  $(\text{t-C}_4\text{H}_9\text{NH})_2\text{SO}_2$  molecule.

## N-(2-BROMO-2-PROPENYLSULPHINYL)-4-METHYLBENZENESULPHONAMIDE



U. SCHUBERT, K. ACKERMANN, G. KRESZE and R. BUSSAS, 1982. Cryst. Struct. Comm., 11, 769-773.

Orthorhombic,  $Pbcn$ ,  $a = 24.548$ ,  $b = 8.053$ ,  $c = 14.111$  Å,  $Z = 8$ . Mo radiation,  $R = 0.082$  for 1043 reflexions.

Molecules are linked by hydrogen bridges between the nitrogen atom and sulphonyl oxygen O(1) (N...O 2.77(1), N-H 1.33(12), O-H 1.50(12) Å). Main bond lengths are S(1)-O(1) 1.46(1), S(1)-N 1.65(1), S(1)-C 1.82(1), S(2)-N 1.66(1), S(2)-O 1.40 and 1.43(1), S(2)-C 1.74(1) Å.

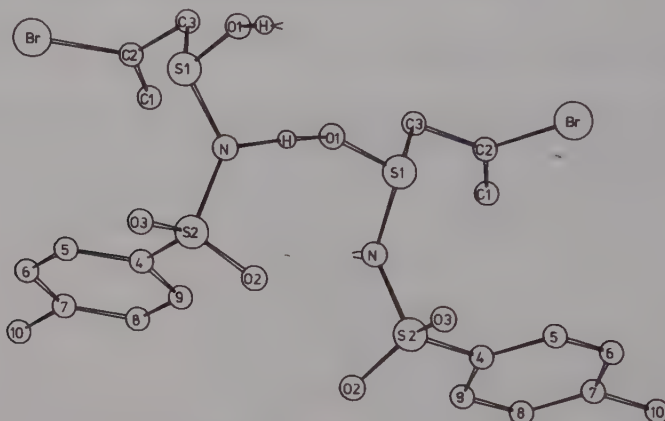


Fig. 1. A view of  $C_{10}H_{12}BrNO_3S_2$  showing the hydrogen bridging.

# BIS(DIPHENYLMETHYLENEAMINE) MONOSULPHIDE

$C_{26}H_{20}N_2S$

M. AVERBUCH-POUCHOT, A. DURIF, A.J. BANISTER, J.A. DURRANT and J. HALFPENNY, 1982. J. Chem. Soc. Dalton, 221-223.

Monoclinic,  $P2_1/a$ ,  $a = 21.323$ ,  $b = 10.260$ ,  $c = 10.271$  Å,  $\beta = 110.50^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.035$  for 2325 reflexions.

The PhCNSNCPh system is largely coplanar, with the remaining phenyl groups twisted  $62.0$  and  $49.2^\circ$  respectively out of the central CNSNC plane. Bond angles at N ( $121.5(2)$ ,  $122.4(2)^\circ$ ) and S ( $94.7(1)^\circ$ ) and bond distances S-N ( $1.675(2)$ ,  $1.676(2)$ ) and C-N ( $1.285(3)$ ,  $1.288(3)$  Å) indicate that the SN bonds are essentially single.

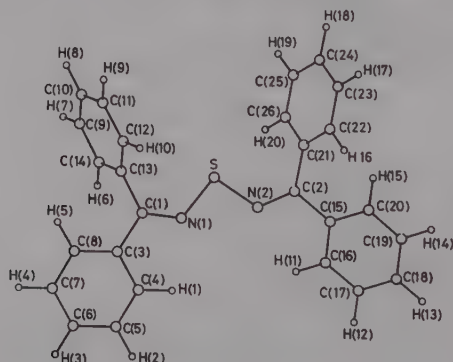


Fig. 1.  $C_{26}H_{20}N_2S$ : the molecular structure.

## CARBON TETRAIODIDE

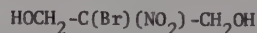
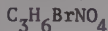


S. POHL, 1982. Z. Krist., 159, 211-216.

Tetragonal,  $I\bar{4}2m$ ,  $a = 6.409$ ,  $c = 9.558 \text{ \AA}$ ,  $Z = 2$ . Mo radiation,  $R = 0.039$  for 88 reflexions.

The molecule is a regular tetrahedron with C-I  $2.155 \text{ \AA}$ . There is a possibility of weak intermolecular I...I interactions.

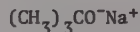
## 2-BROMO-2-NITRO-1,3-PROPANEDIOL



D.S.S. GOWDA and R. RUDMAN, 1982. J. Chem. Phys., 77, 4666-4670.

Monoclinic,  $Cc$ ,  $a = 8.020$ ,  $b = 9.659$ ,  $c = 8.965 \text{ \AA}$ ,  $\beta = 91.00^\circ$ ,  $D_m = 1.845$ ,  $Z = 4$ . Cu radiation,  $R = 0.031$  for 596 reflexions.

The structure is described in terms of a three-dimensional hydrogen-bonded network of interconnected parallel chains with the molecular centres in an approximate tetrahedral arrangement reminiscent of a face-centred unit cell.

SODIUM *t*-BUTOXIDE

J.E. DAVIES, J. KOPF and E. WEISS, 1982. Acta Cryst., B38, 2251-2253.

Trigonal,  $R\bar{3}c$ ,  $a = 19.388$ ,  $c = 43.335 \text{ \AA}$ ,  $Z = 90$ . Radiation given in 1,  $R = 0.170$  for 1730 reflexions.

This structure (Fig. 1), already reported as monoclinic, space group  $Cc$  (1), has been refined again in space group  $R\bar{3}c$ . Bond lengths and angles do not differ significantly from those reported earlier. The  $R\bar{3}c$  cell contains six hexameric and six nonameric  $\text{C}_4\text{H}_9\text{ONa}$  units; the centre of each unit lies on a triad axis. There is considerable rotational disorder associated with the *tert*-butoxy groups.

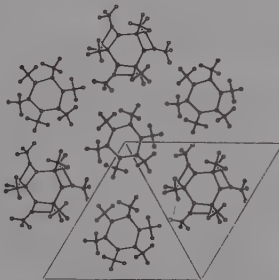
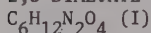


Fig. 1.  $\text{C}_4\text{H}_9\text{NaO}$ : part of the crystal structure projected on to the  $ab$  plane. In this diagram O and Na atoms overlies each other.

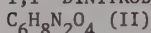
1. Structure Reports, 43B, 68.



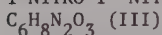
## 2,3-DIMETHYL-2,3-DINITROBUTANE



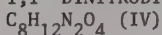
## 1,1'-DINITROBICYCLOPROPYL



## 1-NITRO-1'-NITROBICYCLOPROPYL



## 1,1'-DINITROBICYCLOBUTYL



Y. KAI, P. KNOCHEL, S. KWIATKOWSKI, J.D. DUNITZ, J.M. OTH, D. SEEBACH and H.-O. KALINOWSKI, 1982. *Helv. Chim. Acta*, **65**, 137-161.

I. Triclinic,  $P\bar{1}$ ,  $a = 6.371$ ,  $b = 6.535$ ,  $c = 12.353$  Å,  $\alpha = 103.31^\circ$ ,  $\beta = 79.31^\circ$ ,  $\gamma = 119.84^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.031$  for 739 reflexions (at room temperature).

II. Triclinic,  $P\bar{1}$ ,  $a = 6.300$ ,  $b = 6.380$ ,  $c = 11.851$  Å,  $\alpha = 100.73^\circ$ ,  $\beta = 81.00^\circ$ ,  $\gamma = 118.72^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.035$  for 1997 reflexions (at 95 K).

III. Monoclinic,  $C2/c$ ,  $a = 10.855$ ,  $b = 6.129$ ,  $c = 11.328$  Å,  $\beta = 97.52^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.035$  for 1104 reflexions (at 95 K).

IV. Monoclinic,  $C2/c$ ,  $a = 10.382$ ,  $b = 6.186$ ,  $c = 11.584$  Å,  $\beta = 99.11^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.061$  for 661 reflexions (at 95 K).

IV. Orthorhombic,  $Pbca$ ,  $a = 11.385$ ,  $b = 13.624$ ,  $c = 12.421$  Å,  $Z = 8$ . Mo radiation,  $R = 0.035$  for 1105 reflexions (at 173 K).

All four molecules occur in the gauche conformation, with N-C-C-N torsion angles in the range  $50-70^\circ$ . II and III are isomorphous with twofold crystallographic symmetry; the nitro and nitroso groups of III are disordered. The central C-C bond is long in I (1.575 Å) short in II (1.479 Å), 1.473 in III and 1.517 Å in IV. The C-N bonds are long in I (1.549 Å), short in II (1.488 Å) and 1.548 and 1.551 Å in IV.

## 1,1,1-TRIS(HYDROXYMETHYL)PROPANE (Phase II)



D.S. GOWDA, N. FEDERLEIN and R. RUDMAN, 1982. *J. Chem. Phys.*, **77**, 4659-4665.

Monoclinic,  $P2_1/n$ ,  $a = 8.431$ ,  $b = 9.598$ ,  $c = 9.190$  Å,  $\beta = 98.96^\circ$ ,  $D_m = 1.175$ ,  $Z = 4$ . Cu radiation,  $R = 0.039$  for 1153 reflexions.

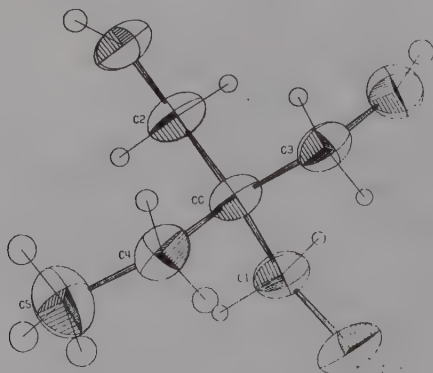


Fig. 1. A view of  $\text{C}_6\text{H}_{14}\text{O}_3$ .

The molecule has a tetrahedral disposition (Fig. 1) with normal dimensions, with O-C-C(C) 111.1-111.5°. The hydroxymethyl groups form a tightly bonded three-dimensional network.

### 2,3-DIPHENYL-2,3-BUTANEDIOL

$C_{16}H_{18}O_2$

F.R. FRONCZEK, M.A. OLIVER and R.D. GANDOUR, 1982. Cryst. Struct. Comm., 11, 1965-1969.

Tetragonal,  $P4_2/n$ ,  $a = 16.300$ ,  $c = 10.279$  Å,  $Z = 8$ . Cu radiation,  $R = 0.062$  for 1015 reflexions.

The molecule (Fig. 1) has approximate  $C_2$  symmetry. Its conformation in the crystal is that in which the hydroxyl groups are gauche (O1-C7-C8-O2 torsion angle 61.4(7)°, the phenyl groups are gauche (C1-C7-C8-C9 -61.4(7)°), and the methyl groups are anti (C15-C7-C8-C16 -172.2(9)°). The major deviation from perfect  $C_2$  molecular symmetry involves slightly different twists of the two phenyl rings relative to the remainder of the molecule. Indicative torsion angles are C2-C1-C7-C8 90.1(8)°, and C14-C9-C8-C7 83.6(8)°. The dihedral angle between the two phenyl planes is 35.1°. The phenyl rings are experimentally planar with normal geometries, having an average C-C bond length of 1.380 Å. Failure to locate the hydroxyl hydrogen atoms is apparently a result of a complex and disordered hydrogen bonding scheme.

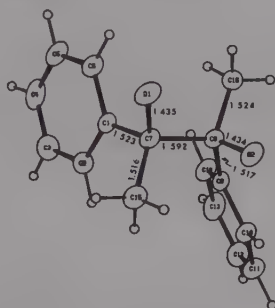


Fig. 1. The  $C_{16}H_{18}O_2$  molecule and bond lengths ( $\sigma$  0.005-0.007 Å).

### 2,4-HEXADIYNE-1,6-DIOL BIS-p-TOLUENESULFONATE

$C_{20}H_{18}O_6S_2$

J.P. AIMÉ, J. LEFEBVRE, M. BERTAULT, M. SCHOTT and J.O. WILLIAMS, 1982. J. Physique, 43, 307-322.

Monoclinic,  $P2_1/c$ ,  $a = 14.745$  (14.630),  $b = 5.086$  (5.133),  $c = 25.738$  (14.845) Å,  $\beta = 91.71$  (118.55)°,  $Z = 4$  (2). Neutron source ( $\lambda = 1.266$  (0.891) Å),  $R = 0.037$  (0.089) for 1869 (1693) reflexions (at 120 K, 221 K).

The low temperature structure consists of monomeric, centrosymmetric molecules (Fig. 1). There are two sets of molecules with different geometries though the differences are slight. Selected average bond lengths are: C(1)≡C(2) 1.213, C(1)-C(3) 1.456, C(3)-O(1) 1.452, O(1)-S(1) 1.596, S(1)-O(2) 1.424, S(1)-O(3) 1.437, S(1)-C(4) 1.753 and C(7)-C(10) 1.505 Å. The toluenesulphonate side groups have few intermolecular contacts and show libration of large amplitude. The high temperature structure shows very large libration on the side groups (without disorder). Bond distances corresponding to those in the 120 K structure are: 1.207, 1.452, 1.442, 1.587, 1.415, 1.431, 1.757 and 1.491 Å respectively. Distortion in the phenyl group is present, bonds C(4)-C(5) and C(7)-C(8) are 1.393 and 1.395 Å at 120 K and 1.372 Å at 221 K.

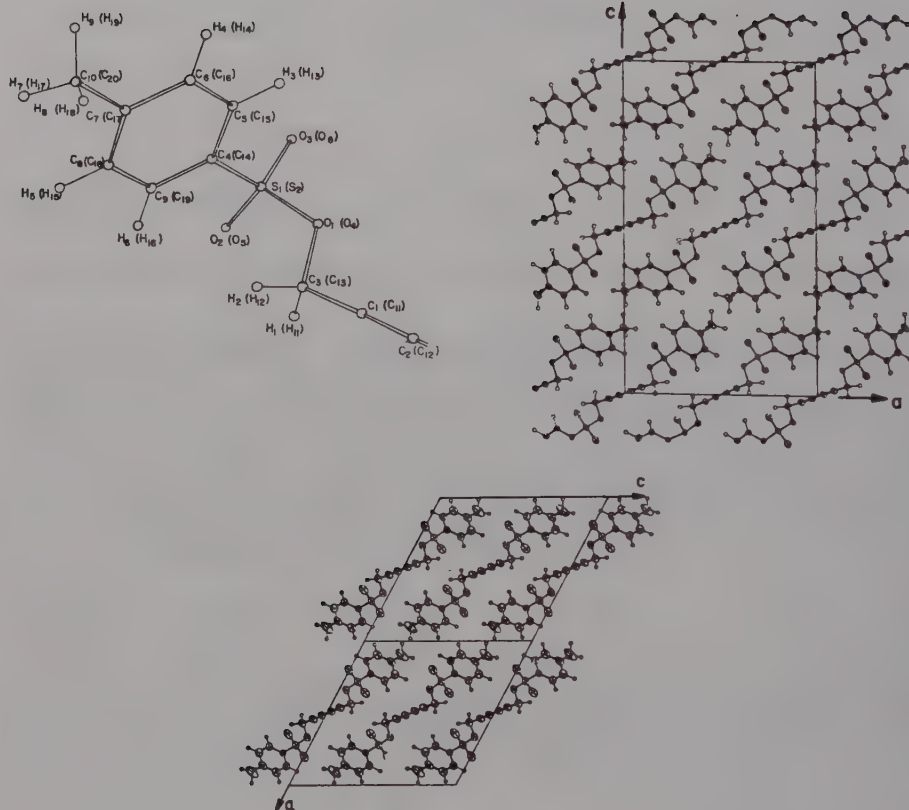


Fig. 1. Site I molecule at 120 K in  $C_{20}H_{18}O_6S_2$  (left), projection of 120 K crystal structure on (101) plane (right), and projection of 221 K structure on (101) plane.

DODECA-5,7-DIYNE-1,12-DIYL BIS-p-TOLUENESULPHONATE (Monomer and Polymer)  
 $C_{26}H_{30}O_6S_2$

D. SIEGEL, H. SIXL, V. ENKELMANN and G. WENZ, 1982. Chem. Phys., 72, 201-212.

#### Monomer

Triclinic,  $P\bar{1}$ ,  $a = 20.60$ ,  $b = 11.79$ ,  $c = 5.19$  Å,  $\alpha = 83.0$ ,  $\beta = 89.2$ ,  $\gamma = 92.7^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.101$  for 1933 reflexions (at 110 K).

#### Polymer

Triclinic,  $P\bar{1}$ ,  $a = 20.01$ ,  $b = 6.02$ ,  $c = 4.91$  Å,  $\alpha = 95.1$ ,  $\beta = 93.7$ ,  $\gamma = 88.7^\circ$ ,  $Z = 1$ . Cu radiation,  $R = 0.098$  for 1047 reflexions (at 110 K).

Views of the monomer and polymer structures are in Fig. 1. Both structures consist of molecular stacks in the (210) plane forming continuous sheets. The polymer lies about inversion centres. Neighbouring diacetylene groups are separated by 5.19 Å. The polymerization process proceeds homogeneously without abrupt change in lattice parameters.

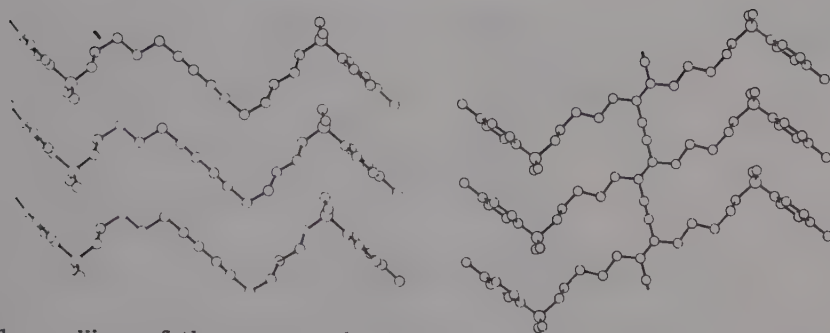


Fig. 1. Views of the monomer and polymer of  $C_{26}H_{30}O_6S_2$ .

#### FUMARONITRILE



D. BRITTON and W.B. GLEASON, 1982. *Cryst. Struct. Comm.*, **11**, 1155-1158.

Monoclinic,  $P2_1/c$ ,  $a = 3.902$ ,  $b = 5.609$ ,  $c = 9.970$  Å,  $\beta = 99.9^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.104$  for 201 reflexions.

The relatively high R-factor is attributed to decomposition of the material in the X-ray beam. The packing is shown in Fig. 1. The molecules lie approximately in layers parallel to the  $(10\bar{2})$  plane. The layers are 3.32 Å apart; the individual molecules are inclined  $8.4^\circ$  with respect to the layers. The most significant interaction appears to be the  $N\dots C(N)$  contact at 3.34 Å shown in Fig. 1.

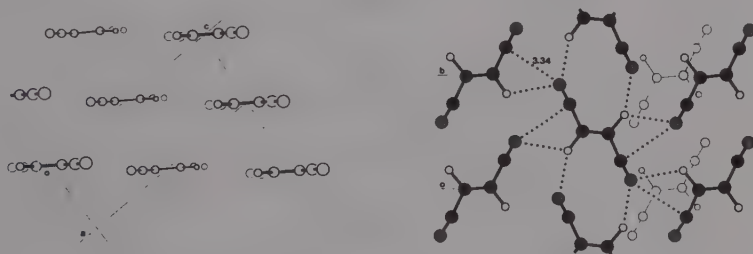


Fig. 1. Two views of the fumaronitrile structure.

#### METHYL 2-CYANO-3-IMINODITHIOBUTYRATE



H. MIYAMAE and H. TAKATO, 1982. *Acta Cryst.*, **B38**, 3166-3168.

Monoclinic,  $P2_1/c$ ,  $a = 8.655$ ,  $b = 12.556$ ,  $c = 8.836$  Å,  $\beta = 119.29^\circ$ ,  $D_m = 1.374$ ,  $Z = 4$ . Mo radiation,  $R = 0.039$  for 802 reflexions.

Apart from methyl hydrogen atoms, the molecule (Fig. 1) is planar. Atom C2 is  $sp^2$  hybridized.

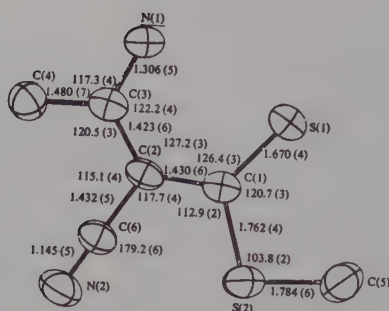


Fig. 1. Bond lengths and angles in  $C_6H_8N_2S_2$ .

# TETRACYANOETHYLENE

$C_6N_4$

U. DRUCK and H. GUTH, 1982. *Z. Krist.*, **161**, 103-110.

Monoclinic,  $P2_1/n$ ,  $z = 2$ . Mo radiation,  $R = 0.035$  for 853 reflexions; neutron radiation,  $R = 0.033$  for 686 reflexions.

Contrary to a previous study (1) a slight disorder is found, such that 3.8-5% of the molecules are rotated about  $90^\circ$  in the molecular plane around the centre of gravity. Molecular dimensions are in Fig. 1.

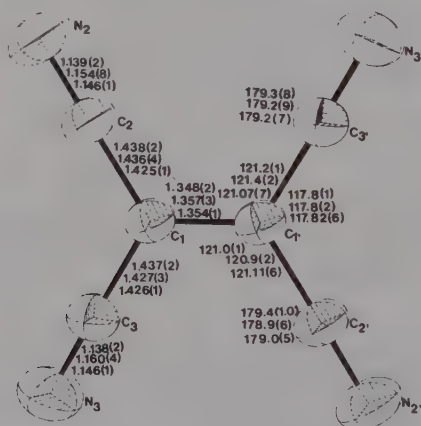


Fig. 1. Molecular dimensions for  $C_6N_4$ . The three values for each dimension correspond to (i) all X-ray data, (ii) high angle X-ray data and (iii) neutron data.

1. *Structure Reports*, **24**, 560.

# p-BENZENEDICARBONITRILE

$C_8H_4N_2$

$p-C_6H_4(CN)_2$

H. GUTH, G. HEGER and U. DRÜCK, 1982. *Z. Krist.*, **159**, 185-190.

Triclinic,  $P\bar{1}$ ,  $a = 3.847$ ,  $b = 6.585$ ,  $c = 7.322$  Å,  $\alpha = 114.5$ ,  $\beta = 93.6$ ,  $\gamma = 96.9^\circ$ ,  $Z = 1$ . Neutron radiation,  $R = 0.052$  for 718 reflexions.

A neutron-diffraction study of a compound described previously by X-ray methods (1). The molecule is planar with C-C (ring) 1.388-1.396(3), C-C (exo) 1.436(4), C≡N 1.148(4), C-H 1.082 and 1.092(4) Å.

1. Structure Reports, 43B, 65; 44B, 46.

#### TETRAMETHYLSUCCINONITRILE



W.B. GLEASON and D. BRITTON, 1982. Cryst. Struct. Comm., 11, 1159-1162.

Monoclinic,  $F2/m$ ,  $a = 12.270$ ,  $b = 9.999$ ,  $c = 6.857$  Å,  $\beta = 93.9^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.066$  for 405 reflexions.

The packing is shown in Fig. 1. The molecules lie in layers parallel to the (010) plane. There is disorder in the structure, with molecules lying in one of two orientations at each site. The anti-parallel nitrile groups are 3.60 Å apart, and there is an N...C(N) contact of 3.25 Å.

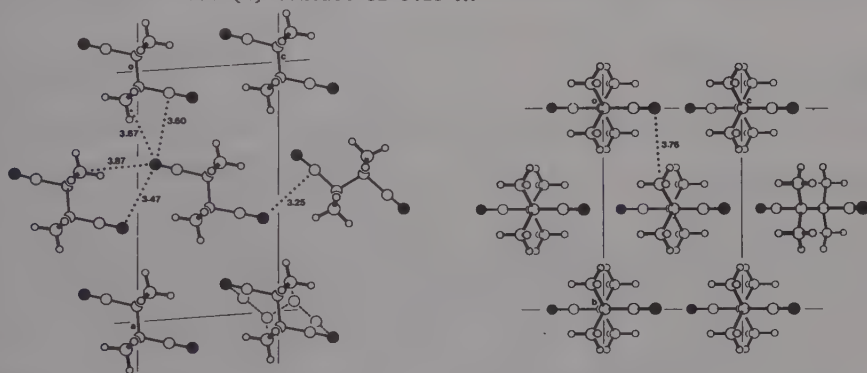
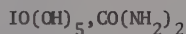
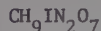


Fig. 1. Views of the structure of tetramethylsuccinonitrile.

#### ORTHOPERIODIC ACID UREA



J. FÁBRY, J. PODLAHOVÁ, J. LOUB and V. LANGER, 1982. Acta Cryst., B38, 1048-1050.

Monoclinic,  $P2_1/c$ ,  $a = 5.175$ ,  $b = 6.870$ ,  $c = 20.480$  Å,  $\beta = 90.96^\circ$ ,  $D_m = 2.60$ ,  $Z = 4$ . Mo radiation,  $R = 0.081$  for 1254 reflexions.

In the crystal structure (Fig. 1) the orthoperiodic acid group forms a distorted octahedron with average I-O 1.88 Å and O-I-O  $90^\circ$  and the urea group is practically planar. The molecules are connected by relatively strong hydrogen bonds to form a three-dimensional network.



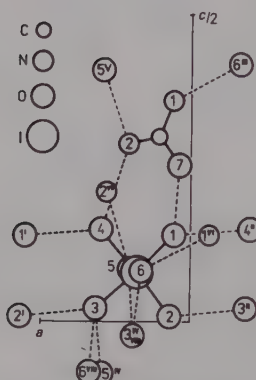


Fig. 1.  $\text{IO}(\text{OH})_5 \cdot \text{CO}(\text{NH}_2)_2$ : schematic projection of the structure in the XZ plane.

# POTASSIUM CYANOGUANIDINE



M.J. BEGLEY and P. HUBBERSTEY, 1982. J. Chem. Research, S, 118-119; M, 1415-1440.

Orthorhombic,  $\text{Pna}2_1$ ,  $a = 7.453$ ,  $b = 5.724$ ,  $c = 11.450 \text{ \AA}$ ,  $D_m = 1.69$ ,  $Z = 4$ .  $R = 0.038$  for 385 reflexions.

The structure (Fig. 1) is built up of alternating sheets of cations and anions which lie parallel to the (001) plane. The three N atoms of each anion coordinate the cation which is six-coordinate ( $\text{K} \dots \text{N}$  2.808-3.012  $\text{\AA}$ ). Hydrogen bonding is limited to one of the three hydrogen atoms on the anion. H(3) links N(4) and N(1) of parallel anionic sheets ( $\text{N}(1) \dots \text{N}(4)$  3.173  $\text{\AA}$ ). The anion has acceptable geometry.

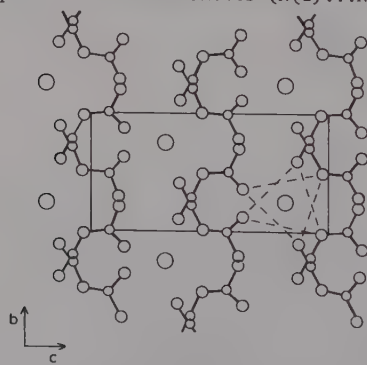
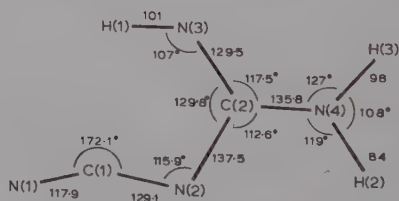


Fig. 1.  $\text{C}_2\text{H}_3\text{KN}_4$ : projection of the structure onto the (100) plane and the geometry of the anion.



# NOXYTHIOLINE



A. TEROL, S. ALBEROLA, B. JEANJEAN, F. SABON and J.C. JUMAS, 1982. Acta Cryst., B38, 636-638.

Orthorhombic,  $\text{Pbca}$ ,  $a = 7.247$ ,  $b = 18.279$ ,  $c = 8.771 \text{ \AA}$ ,  $D_m = 1.36$ ,  $Z = 8$ . Mo radiation,  $R = 0.041$  for 453 reflexions.

Except for O and H, all the atoms lie practically on the N(5),C(4),S(7),N(3) plane (Fig. 1); this plane makes angles of 79.9(7) and 1.3(7)° with the N(3),C(2),O(1) and C(4),N(5),C(6) planes. The molecules are linked into pairs by two N(3)...O(1) hydrogen bonds.

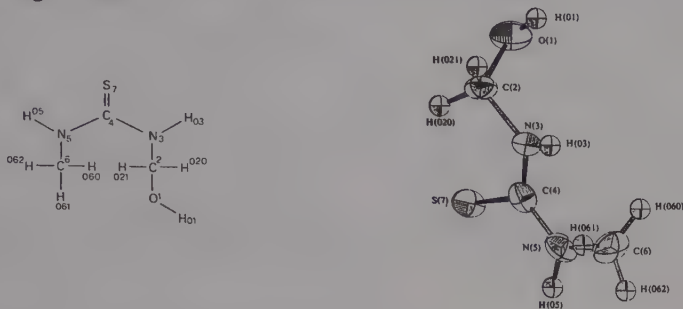
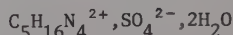
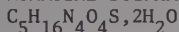


Fig. 1. The noxythioline molecule.

## AGMATINE SULPHATE DIHYDRATE



K. CHANDRASEKHAR, V. PATTABHI and S. RAGHUNATHAN, 1982. *Acta Cryst.*, B38, 2538-2540.

Orthorhombic,  $Pbca$ ,  $a = 22.251$ ,  $b = 7.199$ ,  $c = 15.849$  Å,  $D_m = 1.390$ ,  $Z = 8$ .  $\text{Cu K}\alpha$  radiation,  $R = 0.060$  for 1311 reflexions.

In the crystal structure (Fig. 1) the agmatine chain is essentially planar, packing in layers parallel to ac. Bond lengths and angles are generally as expected. The amine, water and sulphate groups are linked in the crystal by an elaborate network of N-H...O and O-H...O hydrogen bonds.

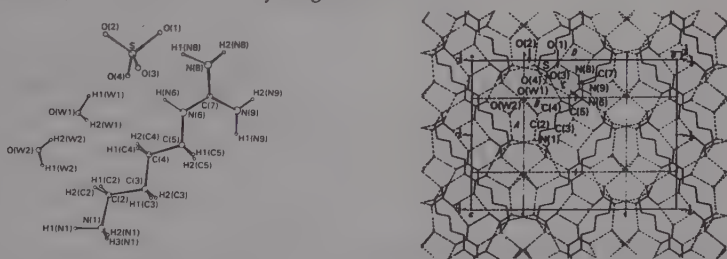
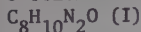
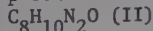


Fig. 1.  $C_5H_{14}N_4, H_2SO_4, 2H_2O$ : perspective view and view of crystal structure projected down  $b$ ; hydrogen bonds are shown by broken lines.

o-TOLYLUREA



p-TOLYLUREA



M.R. CIAJOLO, F. LELJ, T. TANCREDI, P.A. TEMUSSI and A. TUZI, 1982. *Acta Cryst.*, B38, 2928-2930.

I. Monoclinic,  $P2_1/c$ ,  $a = 4.652$ ,  $b = 5.999$ ,  $c = 27.55$  Å,  $\beta = 94.90^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.052$  for 848 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 4.6270$ ,  $b = 5.449$ ,  $c = 31.991$  Å,  $\beta = 99.653^\circ$ ,  $Z = 4$ .  
Cu radiation,  $R = 0.052$  for 959 reflexions.

Both isomers (Fig. 1) have similar molecular parameters with the amide bonds in trans conformation and the aryl rings forming angles of about  $50^\circ$  with the plane of the urea moiety. Extensive networks of intermolecular hydrogen bonds dominate the two crystal packings.

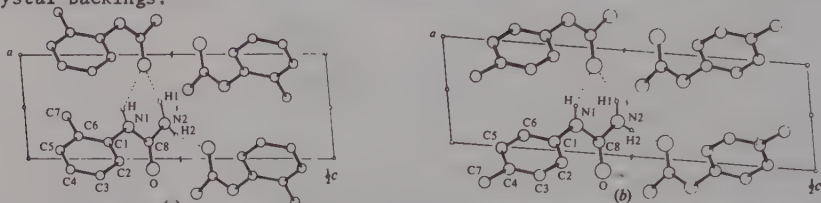
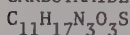


Fig. 1. o-C<sub>6</sub>H<sub>10</sub>N<sub>2</sub>O (I) and p-C<sub>6</sub>H<sub>10</sub>N<sub>2</sub>O (II): mode of packing for (a) I and (b) II along the [010] direction. Some hydrogen bonds are indicated as dashed lines.

#### CARBUTAMIDE (1-BUTYL-3-SULPHANYL UREA)



C.H. KOO, S.I. CHO and Y.H. YEON, 1982. J. Pharm. Soc. Korea, **26**, 9-23.

Orthorhombic, P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, a = 9.257, b = 9.928, c = 15.278 Å, D<sub>m</sub> = 1.28, Z = 4. Cu radiation, R = 0.062 for 575 reflexions.

The atoms forming the urea moiety (Fig. 1) are essentially planar, the largest deviation from the best plane through atoms O(3), N(2), N(3) and C(7) being -0.002 Å for C(7). Other features of the structure include layers of molecules joined by N-H...O hydrogen bonds with distances ranging from 2.745 to 3.100 Å. The bonds include bifurcated hydrogen bonds across the screw axes along a and b.

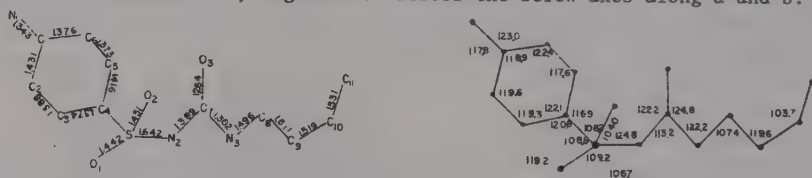


Fig. 1. Bond lengths (Å) and angles (°) in C<sub>11</sub>H<sub>17</sub>N<sub>3</sub>O<sub>3</sub>S.

#### N,N'-DIFORMOHYDRAZIDE (1,2-HYDRAZINEDICARBOXALDEHYDE)



G.A. JEFFREY, J.R. RUBLE, R.K. McMULLAN, D.J. DEFREES and J.A. POPLER, 1982. Acta Cryst., **B38**, 1508-1513.

Monoclinic, P2<sub>1</sub>/c, a = 3.4724, b = 6.1770, c = 8.939 Å, β = 111.92°, Z = 2. Neutron radiation (λ = 1.0442 Å), R = 0.024 for 710 reflexions (at 15 K).

The molecular structure has been the subject of previous investigations (1). Molecular symmetry is  $\bar{1}$  and is close to 2/m with H(N) showing maximum deviation from planarity (0.0102 Å). Molecules are hydrogen bonded in the (10 $\bar{2}$ ) planes (Fig. 1) which are separated by 3.184(5) Å. Thermal motion analysis gives a good fit for the non-hydrogen atoms.

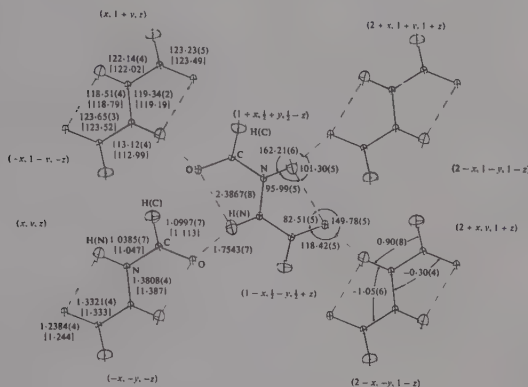


Fig. 1. The crystal structure of  $N,N'$ -diformohydrazide. Values in square brackets are corrected for thermal motion. Broken lines indicate hydrogen bonds.

1. Structure Reports, 22, 611; 44B, 53.

$N$ -METHYL- $N$ -NITROSOUREA

$C_2H_5N_3O_2$  (I)

$CH_3(NO)NCONH_2$

$N,N'$ -DIMETHYL- $N$ -NITROSOUREA

$C_3H_7N_3O_2$  (II)

$CH_3(NO)NCONHCH_3$

2-NITROSO-2-AZABICYCLO[2.2.2]OCTAN-3-ONE

$C_7H_{10}N_2O_2$  (III)

$N$ -METHYL- $N$ -NITROSO- $p$ -NITROBENZAMIDE

$C_8H_7N_3O_4$  (IV)

K. PROUT, J. FAIL, S. HERNANDEZ-CASSOU and F.M. MIAO, 1982. Acta Cryst., B38, 2176-2181.

I. Monoclinic,  $P2_1/n$ ,  $a = 5.302$ ,  $b = 5.617$ ,  $c = 15.442$  Å,  $\beta = 90.09^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.041$  for 725 reflexions.

II. Monoclinic,  $P2_1/a$ ,  $a = 8.412$ ,  $b = 9.953$ ,  $c = 7.435$  Å,  $\beta = 115.09^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.042$  for 809 reflexions.

III. Orthorhombic,  $Pnam$ ,  $a = 15.634$ ,  $b = 6.966$ ,  $c = 6.698$  Å,  $Z = 4$ . Mo radiation,  $R = 0.071$  for 540 reflexions.

IV. Triclinic,  $P\bar{1}$ ,  $a = 7.116$ ,  $b = 8.414$ ,  $c = 8.462$  Å,  $\alpha = 103.24$ ,  $\beta = 103.11$ ,  $\gamma = 97.98^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.042$  for 679 reflexions.

The nitrosamide residue  $R-C(O)-N(NO)-R'$  is best regarded as a planar group in all four compounds (Fig. 1). The nitroso  $N-O$  bond lengths are: 1.231(2) (I), 1.227(2) (II), 1.227(5) (III) and 1.218(4) Å (IV). Cohesion of the crystal structures is effected by hydrogen-bonding networks in I and II and by van der Waals packing forces in III and IV.

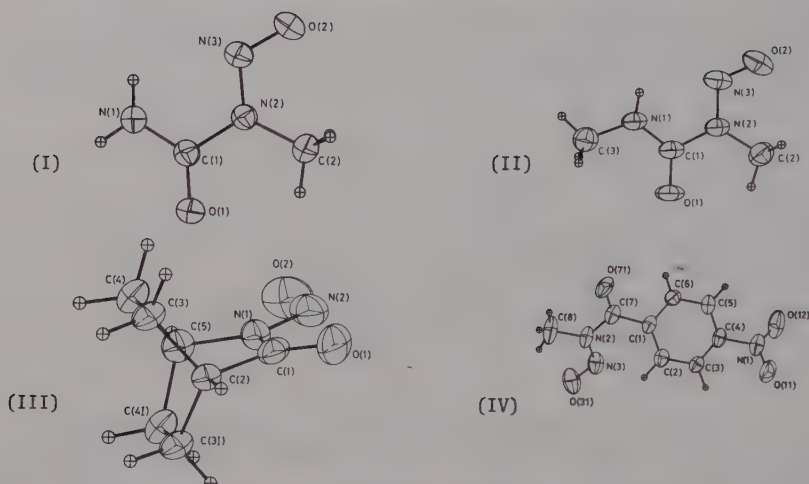


Fig. 1. The molecular structures of  $C_2H_5N_3O_2$  (I),  $C_3H_7N_3O_2$  (II),  $C_7H_{10}N_2O_2$  (III) and  $C_8H_7N_3O_4$  (IV).

#### TETRAFORMOXYDIAZIDE



T. OTTERSEN, J. ALMLÖF and J. CARLÉ, 1982. *Acta Chem. Scand.*, **A36**, 63-68.

Tetragonal,  $I\bar{4}2m$ ,  $a = 6.443$ ,  $c = 7.475$  Å,  $Z = 2$ . Mo radiation,  $R_w = 0.038$  for 730 reflexions (at 110 K).

This structural investigation was undertaken to obtain more information about electron density distribution in the N-C=O and N-N moieties than was available from an earlier study (1). The torsional angle around the N-N bond is crystallographically determined at  $90^\circ$  with planar configurations around the N atoms. The bond lengths differ widely (Fig. 1) from those reported earlier (1), due to the low overdetermination ratio and the refinement used in the earlier investigation.

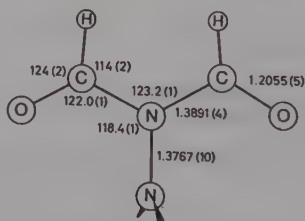


Fig. 1. Bond distances and angles in  $C_4H_4N_2O_4$ .

1. *Structure Reports*, **40B**, 64.

#### 2,2'-DINITROXYDIETHYLNITRAMINE



A. WILKINS and R.W.H. SMALL, 1982. *Acta Cryst.*, **B38**, 488-490.

Monoclinic,  $P2_1/c$ ,  $a = 9.06$ ,  $b = 9.15$ ,  $c = 12.33$  Å,  $\beta = 109.94^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.039$  for 909 reflexions.

Figure 1 compares the 2,2'-dinitroxydiethylnitramine molecule with that found in a previously determined polymorph (1). Differences occur by rotation about the C(1)-N(2) and C(3)-O(3) bonds but the conformation about C(1)-C(3) is unchanged with attached groups in the gauche conformation. Corresponding distances and angles in both halves of the molecule do not differ appreciably and are close to those given in (1). The atoms O(1)O(2)N(1)N(2)C(1)C(2) are planar within 0.13 Å; in the O(6)O(8)N(4)O(4)C(4)C(2) group, C(2) is 0.574 Å from the nitro-group plane; in O(5)O(7)N(3)O(3)C(3)C(1), C(3) is 0.061 Å from the nitro-group plane, with C(1) displaced 1.336 Å on the opposite side. The shortest intermolecular contact is 3.12 Å between O(6) and N(3).

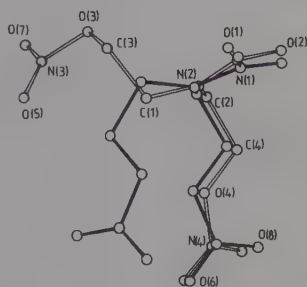
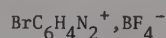
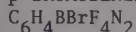


Fig. 1. Comparison of the 2,2'-dinitroxydiethylnitramine molecule in two different polymorphs.

1. Structure Reports, 44B, 56.

p-BROMOBENZENEDIAZONIUM TETRAFLUOROBORATE



K. SASVARI, H. HESS and W. SCHWARZ, 1982. Cryst. Struct. Comm., 11, 781-786.

Monoclinic,  $P2_1/c$ ,  $a = 5.503$ ,  $b = 10.361$ ,  $c = 18.113$  Å,  $\beta = 116.57^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.070$  for 1739 reflexions (at 143 K).

In this structure (Fig. 1) the main bond lengths are  $\text{N}=\text{N}$  1.082(8),  $\text{C}-\text{N}$  1.404(8),  $\text{C}-\text{Br}$  1.885(6) Å with  $\text{C}-\text{N}=\text{N}$  177.6(7)°. The  $\text{BF}_4^-$  ions make contacts ( $\text{F}\cdots\text{N}$  2.82(1) Å) with adjacent cations.

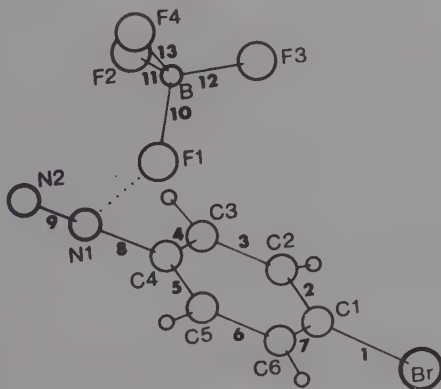
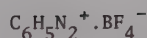
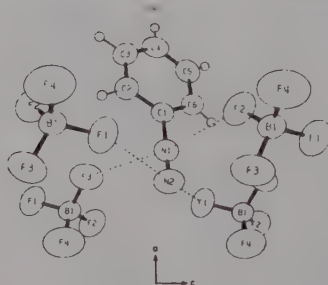


Fig. 1. The  $\text{BrC}_6\text{H}_4\text{N}_2^+, \text{BF}_4^-$  structure.



$$\text{C}_6\text{H}_5\text{BF}_4\text{N}_2$$


Monoclinic,  $P2_1/a$ ,  $a = 17.347$ ,  $b = 8.396$ ,  $c = 5.685$  Å,  $\beta = 92.14^\circ$ ,  $D_m = 1.53$ ,  $Z = 4$ .  
Cu radiation,  $R = 0.063$  for 1346 reflexions.


$$\text{C}_7\text{H}_8\text{N}_2\text{OS}$$
[illegible]

1	1.455(4)	4	1.478(6)	7	1.376(8)	10	1.370(7)
2	1.542(4)	5	1.414(6)	8	1.392(8)	11	1.391(7)
3	1.303(5)	6	1.382(9)	9	1.362(10)		
1,2	121.2(2)	3,5	116.7(4)	5,11	120.5(6)	8,9	119.2(5)
2,3	133.2(3)	4,5	120.7(4)	6,7	119.6(5)	9,10	120.7(5)
3,4	122.6(4)	5,6	120.3(5)	7,8	120.8(5)	10,11	120.5(5)
						6,11	119.2(5)

$$\text{C}_{10}\text{H}_{12}\text{N}_4\text{OS}, 0.5\text{H}_2\text{O}$$


Triclinic,  $P\bar{1}$ ,  $a = 11.806$ ,  $b = 14.336$ ,  $c = 7.349$  Å,  $\alpha = 95.61$ ,  $\beta = 94.83$ ,  $\gamma = 96.89^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.064$  for 1960 reflexions.

The unit cell contains two crystallographically-independent molecules, differing mainly in the orientation of the phenyl rings. Figure 1 shows the atomic rotation, bond distances and valency angles for each of these molecules. The molecules of the thiosemicarbazone and water are linked by a three-dimensional framework of hydrogen bonds (Fig. 1).

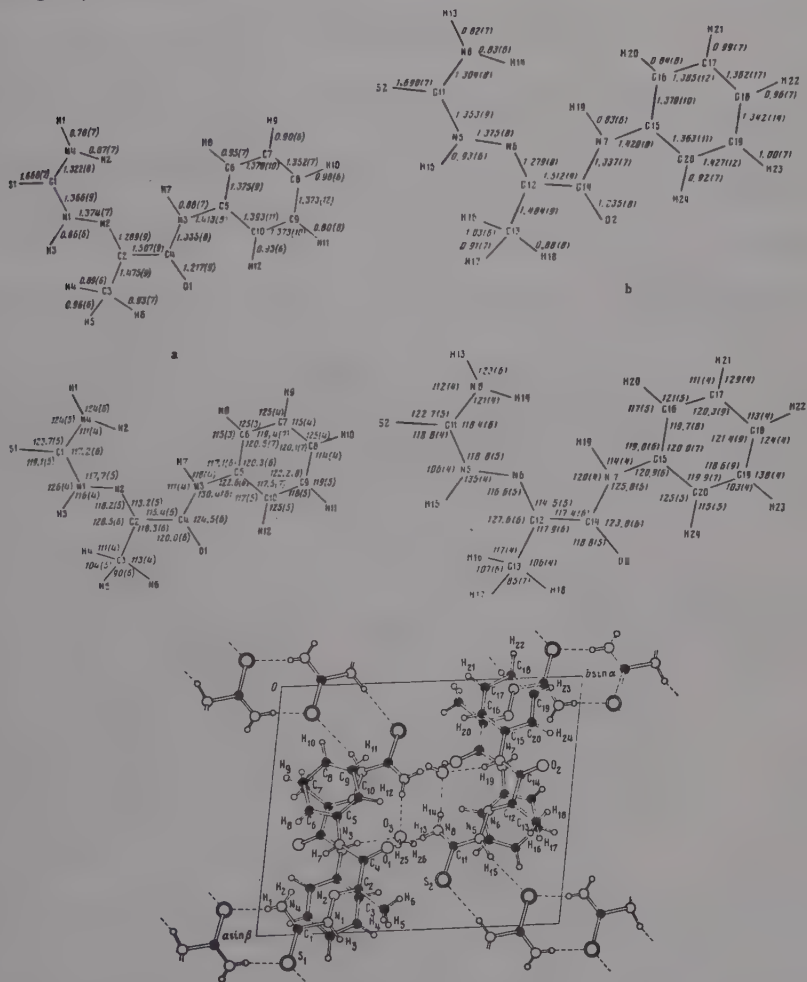
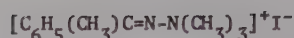
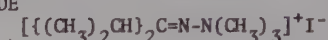


Fig. 1. Valence distances and angles in  $C_{10}H_{12}N_4OS$  (top), and projection of crystal structure on the (001) plane (bottom).

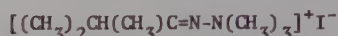
ACETOPHENONE N,N,N-TRIMETHYLHYDRAZONIUM IODIDE

 $C_{11}H_{17}IN_2$  (I)

2,4-DIMETHYL-3-PENTANONE N,N,N-TRIMETHYLHYDRAZONIUM IODIDE

 $C_{10}H_{23}IN_2$  (II)

3-METHYL-2-BUTANONE N,N,N-TRIMETHYLHYDRAZONIUM IODIDE

 $C_8H_{19}IN_2$  (III)

S. ARSENIYADIS, A. LAURENT, P. MISON, R. FAURE and H. LOISELEUR, 1982. Acta Cryst., B38, 2185-2190.

I. Tetragonal,  $P4_12_1$ ,  $a = 10.703$ ,  $c = 22.966$  Å,  $Z = 8$ . Ag radiation,  $R = 0.030$  for 1242 reflexions.

II. Orthorhombic,  $Pnma$ ,  $a = 12.265$ ,  $b = 9.749$ ,  $c = 23.918$  Å,  $Z = 8$ . Mo radiation,  $R = 0.057$  for 1492 reflexions.

III. Orthorhombic,  $Pnma$ ,  $a = 12.769$ ,  $b = 7.857$ ,  $c = 11.485$  Å,  $Z = 4$ . Ag radiation,  $R = 0.032$  for 859 reflexions.

In compound I, atoms N(1), N(2), C(1), C(2) and C(3) of the cation lie in a plane which acts as a mirror plane except for the phenyl ring; in compound II there are two independent cations which have the same structure, each with  $m$  symmetry; in compound III the cation also has  $m$  symmetry (Fig. 1). These structure determinations allow the shielding effects observed in the  $^{13}C$  NMR spectrum to be rationalized. The shielding of the syn methyl carbon in I and III is explained by means of steric interactions, to which are also attributed the shielding of the syn and anti methine C atoms in II.

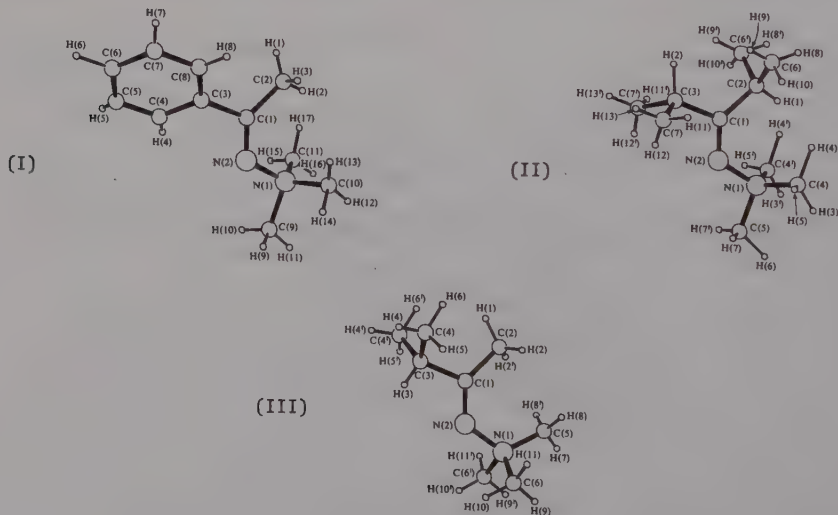


Fig. 1. The molecular structures of  $C_{11}H_{17}IN_2$  (I),  $C_{10}H_{23}IN_2$  (II) and  $C_8H_{19}IN_2$  (III).

$\alpha, \alpha'$ -DIMETHYL- $\alpha, \alpha'$ -AZINODI-*p*-CRESOL MONOHYDRATE  
 $C_{16}H_{16}N_2O_2 \cdot H_2O$

M.C. GARCÍA-MINA, F. ARRESE, M. MARTÍNEZ-RIPOLL, S. GARCÍA-BLANCO and J.L. SERRANO, 1982. *Acta Cryst.*, B38, 2726-2728.

Monoclinic,  $P2_1/c$ ,  $a = 15.871$ ,  $b = 8.300$ ,  $c = 11.496$  Å,  $\beta = 94.42^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.068$  for 1088 reflexions.

A torsional angle of  $148(1)^\circ$  through the azine chain makes the molecule non-planar (Fig. 1). Bond lengths and angles are generally as expected, but the N-N bond length ( $1.417(7)$  Å) is significantly less than the expected  $N(sp^3)-N(sp^3)$  bond length ( $1.449$  Å). No face-to-face close-packing of molecules occurs. The molecules are held together through O-H...O and O-H...N hydrogen bonds involving an N atom of the azine chain, both molecular hydroxy groups and the water molecule.

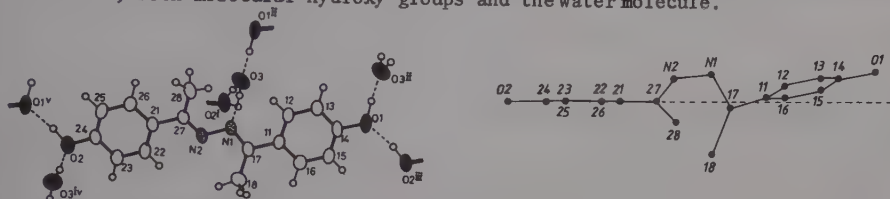


Fig. 1.  $C_{16}H_{16}N_2O_2 \cdot H_2O$ : perspective drawing of the molecule and its nearest neighbours (dashed lines represent hydrogen bonds) and a schematic drawing showing departures from planarity.

DIETHYL 2-(*o*-METHYLPHENYLHYDRAZONO)-3-OXOPENTANEDIOATE  
 $C_{16}H_{20}N_2O_5$  (I)

DIETHYL 2-(*p*-NITROPHENYLHYDRAZONO)-3-OXOPENTANEDIOATE (MONOCLINIC AND ORTHORHOMBIC FORMS)  
 $C_{15}H_{17}N_2O_7$  (II)

M.G.B. DREW, B. VICKERY and G.R. WILLEY, 1982. *Acta Cryst.*, B38, 1530-1535.

I. Triclinic,  $P\bar{1}$ ,  $a = 8.15$ ,  $b = 11.34$ ,  $c = 9.46$  Å,  $\alpha = 102.3$ ,  $\beta = 104.5$ ,  $\gamma = 95.0^\circ$ ,  $D_m = 1.32$ ,  $Z = 2$ . Mo radiation,  $R = 0.074$  for 1563 reflexions.

IIa. Monoclinic,  $P2_1$ ,  $a = 4.895$ ,  $b = 12.934$ ,  $c = 13.365$  Å,  $\beta = 93.6^\circ$ ,  $D_m = 1.37$ ,  $Z = 2$ . Mo radiation,  $R = 0.062$  for 822 reflexions.

IIb. Orthorhombic,  $P22_12_1$ ,  $a = 4.88$ ,  $b = 13.13$ ,  $c = 26.56$  Å,  $D_m = 1.38$ ,  $Z = 4$ . Mo radiation,  $R = 0.065$  for 999 reflexions.

There is an intramolecular hydrogen bond in all three molecules ( $N(7) \dots O(11)$  is  $2.60$  (I),  $2.64$  (IIa),  $2.64$  (IIb) Å) but no intermolecular hydrogen bonds. In I (Fig. 1) the  $C(7)-C(3)-C(4)-N(7)$  torsion angle is  $-1.7^\circ$  and the whole molecule is more planar than those of IIa and IIb (Fig. 1): the maximum deviation of  $N(7), N(8), C(9), C(10), C(20)$  from the phenyl ring plane is  $0.1$  Å in I and is more than  $0.5$  Å in IIa and IIb; the  $C(9), C(10), O(11), O(12), C(13)$  plane intersects the ring plane at  $7.2$  (I),  $27.6$  (IIa),  $24.6^\circ$  (IIb). The  $C(9), C(20), O(21), C(22)$  plane makes angles of  $10.2$  (I),  $8.5$  (IIa) and  $5.8^\circ$  (IIb) with the phenyl ring and this plane is almost perpendicular to the  $C(22), C(23), O(24), O(25)$  plane in all three molecules. Some bond lengths are (in order I, IIa, IIb),  $C(4)-N(7)$   $1.412(6)$ ,  $1.375(10)$ ,  $1.380(15)$ ;  $N(7)-N(8)$   $1.302(5)$ ,  $1.325(9)$ ,  $1.305(12)$ ;  $N(8)-C(9)$   $1.313(5)$ ,  $1.292(10)$ ,  $1.339(11)$  Å. IIa and IIb have almost identical molecular structures and similar packing but packing is less efficient along  $z$  than along  $x$  and  $y$  thus accounting for the different crystal structures.

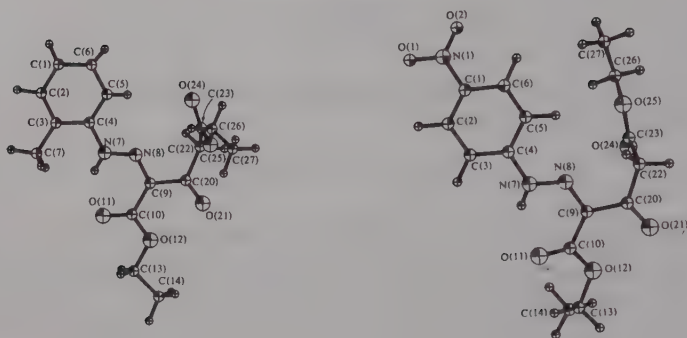


Fig. 1. Views of the  $C_{16}H_{20}N_2O_5$  (I) and  $C_{15}H_{17}N_3O_7$  (IIa) molecules.

1,5-BIS(2,6-DIMETHYLPHENYL)-3-NITROFORMAZAN  
 $C_{17}H_{19}N_5O_2$

E. DIJKSTRA, A.T. HUTTON, H.M.N.H. IRVING and L.R. NASSIMBENI, 1982. *Acta Cryst.*, B38, 535-539.

Monoclinic,  $P2_1/n$ ,  $a = 16.419$ ,  $b = 7.497$ ,  $c = 13.443$  Å,  $\beta = 90.69^\circ$ ,  $D_m = 1.29$ ,  $Z = 4$ . Mo radiation,  $R = 0.063$  for 1143 reflexions.

There is extensive delocalisation of  $\pi$  electrons in the planar N-N-C-N-N chain which adopts the closed-ring syn,s-cis configuration relative to the formal C=N and C-N bonds with the imino proton forming an intramolecular hydrogen bridge N(1)...N(5) 2.72(1) Å (Fig. 1). The aryl groups are twisted  $26^\circ$  and  $36^\circ$  and the nitro-group  $23^\circ$  out of the formazan ring plane. Some dimensions are N(1)-N(2) 1.298(5), N(4)-N(5) 1.301(5), C(3)-N(2) 1.346(6), C(3)-N(4) 1.322(6), C(3)-N(3) 1.484(6) Å, N(2)-C(3)-N(4)  $136^\circ$ .

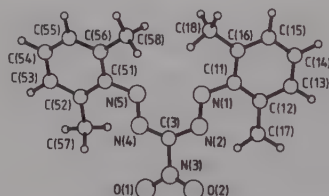


Fig. 1. The 1,5-bis(2,6-dimethylphenyl)-3-nitroformazan molecule.

GLYOXIME  
 $C_2H_4N_2O_2$

G.A. JEFFREY, J.R. RUBLE and J.A. POPLE, 1982. *Acta Cryst.*, B38, 1975-1980.

Monoclinic,  $P2_1/c$ ,  $a = 3.745$ ,  $b = 4.398$ ,  $c = 10.852$  Å,  $\beta = 92.41^\circ$ ,  $Z = 2$ . Neutrons ( $\lambda = 1.0470$ ),  $R = 0.029$  for 772 reflexions (at 9 K).

The molecule (Fig. 1), whose crystal structure has been previously determined (1), has crystallographic  $\bar{1}$  symmetry and is planar except for the oxime H which is displaced 0.263(1) Å from the mean plane of the other atoms. Molecules are hydrogen bonded into buckled layers parallel to (100) with 3.745(1) Å separating the layers. Adjacent rows of hydrogen-bonded glyoxime molecules in the same layer have their planes at approximately  $90^\circ$  to each other. Ab initio molecular-orbital calculations show that the conformation observed in the crystal (anti-anti-anti) is that of minimum energy calculated for the isolated molecule at rest.

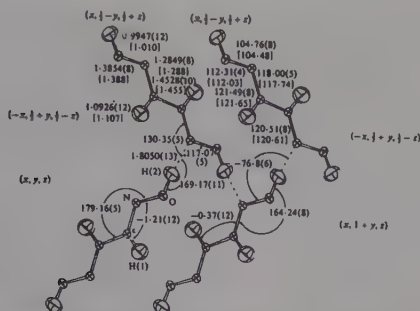
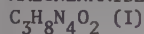


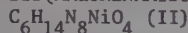
Fig. 1. Molecular dimensions (those in square brackets are corrected for thermal motion) in glyoxime.

# 1. Structure Reports, 31B, 53.

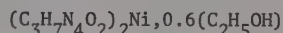
## MALONENAMIDE OXIME



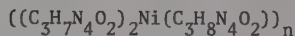
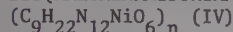
## BIS(MALONENAMIDOXIMATO)NICKEL(II)



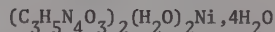
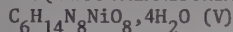
## BIS(MALONENAMIDOXIMATO)NICKEL(II) ETHANOL SOLVATE



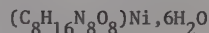
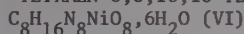
## BIS(MALONENAMIDOXIMATO)(MALONENAMIDOXIME)NICKEL(II)



## BIS(MESOXALAMIDOXIMATO)DIAQUANICKEL(II) TETRAHYDRATE



## 3,4,11,13-TETRAAMINO-1,7,9,15-TETRAOXA-2,6,10,14-TETRAAZA-2,5,10,13-CYCLOHEXADECATETRAEN-8,8,16,16-TETROL HEXAHYDRATE



H. ENDRES and N. KADKHODAI-ELYADERANI, 1982. Z. Naturforsch., 37b, 1255-1265.

I. Monoclinic, C2/c,  $a = 8.686$ ,  $b = 9.520$ ,  $c = 14.306$  Å,  $\beta = 96.79^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.066$  for 1116 reflexions.

II. Orthorhombic, Pnam,  $a = 8.357$ ,  $b = 9.385$ ,  $c = 14.794$  Å,  $Z = 4$ . Mo radiation,  $R = 0.045$  for 1384 reflexions.

III. Orthorhombic, C2cm,  $a = 7.639$ ,  $b = 12.890$ ,  $c = 15.261$  Å,  $Z = 4$ . Mo radiation,  $R = 0.053$  for 584 reflexions.

IV. Monoclinic, C2/c,  $a = 16.014$ ,  $b = 8.026$ ,  $c = 15.193$  Å,  $\beta = 119.34^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.035$  for 1740 reflexions.

V. Monoclinic, P2<sub>1</sub>/a,  $a = 7.295$ ,  $b = 16.063$ ,  $c = 8.226$  Å,  $\beta = 116.04^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.028$  for 866 reflexions.

VI. Monoclinic, P2<sub>1</sub>/n,  $a = 8.609$ ,  $b = 11.714$ ,  $c = 10.760$  Å,  $\beta = 111.91^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.029$  for 2244 reflexions.

I exists in a transoid configuration (Fig. 1) and molecules are linked together by hydrogen bonds (O-H...O 2.745(4), O-H...N 2.688(4) Å). Principal bond lengths are: C(2)-C(1), C(3) 1.506(5), C(1)-N(1), C(3)-N(3) 1.273(5), C(1)-N(2), C(3)-N(4) 1.350(5), N(1)-O(1), N(3)-O(2) 1.436(4) Å. The Ni in II is square-planar coordinated with Ni-N 1.886(3) Å. The molecule has C<sub>s</sub> symmetry, adopts overall a boat form, and



contains two intramolecular O-H...O bonds. III adopts the same overall shape as II except the molecular symmetry is  $C_2$ . In IV the Ni coordination is extended to octahedral bridging malonenamide oxime moieties. The molecular symmetry is  $C_i$  with Ni-N(eq.) 2.059(2) and Ni-N(axial) 2.295(3) Å. In V the Ni is also octahedrally coordinated with molecular  $C_i$  symmetry (Ni-N 2.097(3), Ni-O 2.115(5) Å). The overall conformation is a flattened chair produced by bending the two halves of the molecule at the N(1)...N(2) line by some  $10^\circ$ . The Ni in the centrosymmetric VI is square-planar coordinated with Ni-N 1.872(2) Å.

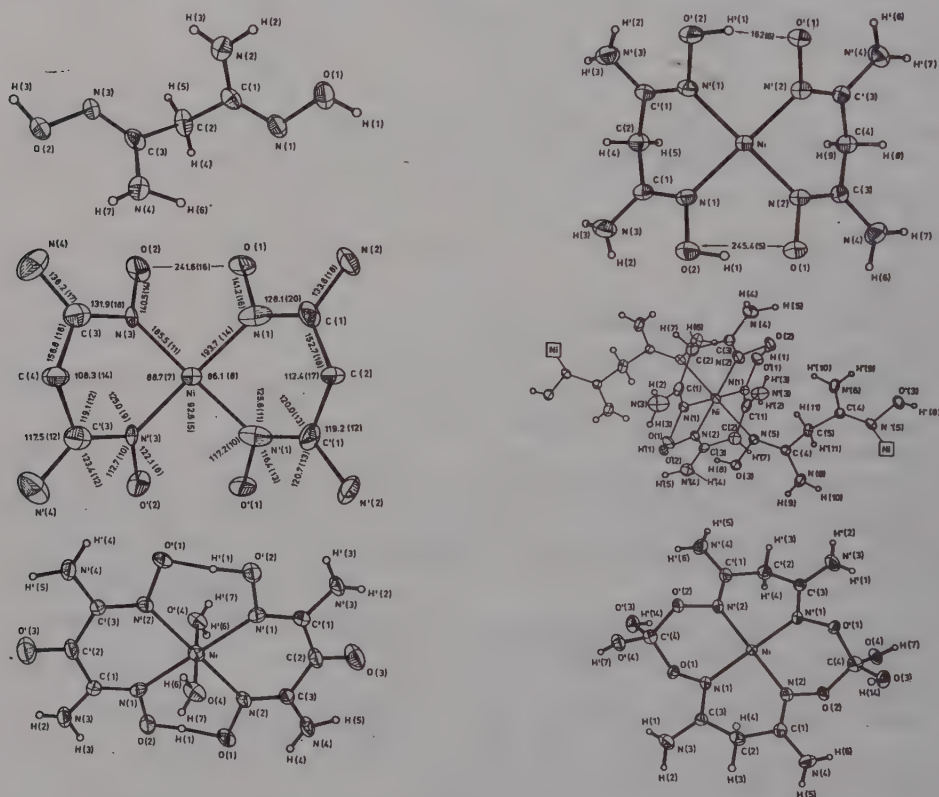


Fig. 1. Views of the oxime  $C_3H_8N_4O_2$  (I) (top left) and the nickel complexes (II) (top right), (III) (centre left), (IV) (centre right), (V) (bottom left), and (VI) (bottom right).

1,4-DIFLUORO-1,1,4,4-TETRANITRO-2,3-DINITROOXYBUTANE  
 $C_4H_2F_2N_6O_{14}$

H.L. AMMON and S.K. BHATTACHARJEE, 1982. *Acta Cryst.*, B38, 2718-2721.

Monoclinic,  $P2_1/c$ ,  $a = 7.6975$ ,  $b = 5.7890$ ,  $c = 14.722$  Å,  $\beta = 101.74^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.054$  for 811 reflexions.

The molecule (Fig. 1) lies on a centre of symmetry and has an overall structure and conformation similar to that of 1,4-difluoro-1,1,4,4-tetranitro-2,3-butanediol (1). The O(5)-N(3) distance of 1.515(5) Å, which is substantially longer than other O-N single bonds, may account for the facile hydrolysis of the nitrate ester linkage. There are no short intermolecular contacts that would account for the exceptional crystal density, calculated to be 2.05.

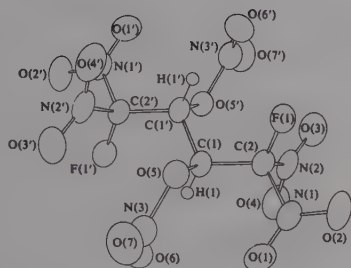
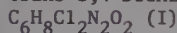


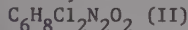
Fig. 1.  $C_6H_2F_2N_6O_{14}$ : perspective view of the molecule and bond lengths and angles in the asymmetric unit.

1. Structure Reports, 45B, 47.

trans-1,4-DICHLORO-1,4-DINITROSOCYCLOHEXANE



cis-1,4-DICHLORO-1,4-DINITROSOCYCLOHEXANE



FANG MING MIAO, D. CHANTRY, T. HARPER and D.C. HODGKIN, 1982. *Acta Cryst.*, B38, 3152-3155.

I. Orthorhombic, *Pcab*,  $a = 8.777$ ,  $b = 10.057$ ,  $c = 10.133$  Å,  $D_m = 1.56$ ,  $Z = 4$ . Mo radiation,  $R = 0.033$  for 341 reflexions.

II. Orthorhombic, *Pmmn*,  $a = 9.5$ ,  $b = 7.15$ ,  $c = 6.21$  Å,  $Z = 2$ . 198 reflexions (Film data, projection study.)

The trans-isomer (I) consists of C-nitroso monomers lying on inversion centres (Fig. 1) with C-N(O) 1.505(7), (C)N-O 1.139(5) Å and C-N-O 116.5(6)°; the shortest intermolecular O...Cl contact is 3.638(5) Å; the torsion angle Cl-C-N-O is 2.3°. The cis-isomer (II) consists of internal nitroso dimers with the cyclohexane ring in the boat form; the nitroso dimer has the *mm* symmetry of the space group; N-N 1.31, N-O 1.33 and C-N 1.53 Å, N-N-O 121.5, O-N-C 126 and N-N-C 112.5°.

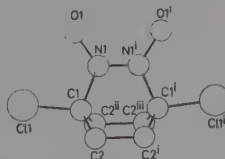
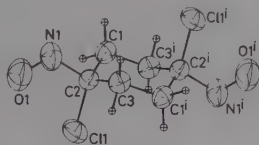
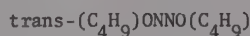
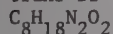


Fig. 1. The trans- (left) and cis- (right) isomers of  $C_6H_8Cl_2N_2O_2$ .

trans-DI-t-BUTYL HYPONITRITE



C.A. OGLE, K.A. VANDERKOOI, G.D. MENDENHALL, V. LORPRAYOON and B.C. CORNILSEN, 1982. *J. Am. Chem. Soc.*, 104, 5114-5119.

Monoclinic,  $P2_1/c$ ,  $a = 5.739$ ,  $b = 10.870$ ,  $c = 8.762$  Å,  $\beta = 108.13^\circ$ ,  $D_m = 1.05$ ,  $Z = 2$ . Mo radiation,  $R = 0.0595$  for 497 reflexions (at  $-50^\circ\text{C}$ ).

The molecule (Fig. 1) possesses crystallographic  $C_i$  symmetry. Important structural parameters:  $N=N$  1.252(6),  $N-O$  1.380(6),  $O-C$  1.471(7) Å, and  $N=N-O$   $106.5(3)^\circ$ .

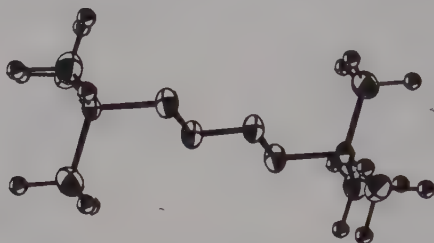
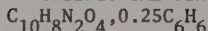


Fig. 1. The  $\text{trans}-(\text{C}_4\text{H}_9)\text{ONNO}(\text{C}_4\text{H}_9)$  molecule.

#### $\alpha$ -FURILDIOXIME BENZENE SOLVATE



R.E. STENKAMP, L.H. JENSEN, T.B. MURPHY and N.J. RCSE, 1982. Acta Cryst., B38, 1169-1172.

Monoclinic,  $P2_1/c$ ,  $a = 12.426$ ,  $b = 22.583$ ,  $c = 8.208$  Å,  $\beta = 90.10^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.058$  for 2032 reflexions.

Both independent furildioxime molecules are in the  $\alpha$  conformation (Fig. 1). Bond lengths and angles are consistent with those in other structures containing furil and dioxime moieties e.g.  $N-O$  1.387-1.408, average 1.399 Å. All four O-H groups in the asymmetric unit participate in hydrogen bonds with the four N atoms serving as acceptors. In the benzene ring the average C-C distance is 1.356 Å and the average bond angle  $120.0^\circ$ . Van der Waals interactions account for the lack of complete rotational disorder in the benzene molecule.

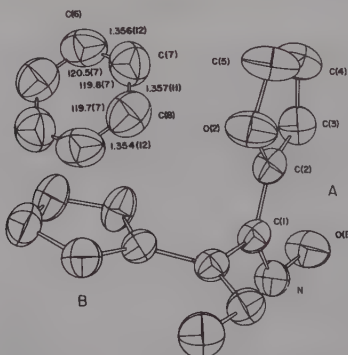


Fig. 1.  $\text{C}_{10}\text{H}_8\text{N}_2\text{O}_4 \cdot 0.25\text{C}_6\text{H}_6$ : a perspective view showing bond lengths and angles for the benzene ring.

E,E-3-(p-TOLYLIMINO)-2-BUTANONE OXIME

$C_{11}H_{14}N_2O$  (I)

E,Z-4-(p-TOLYLIMINO)-2,3-PENTANEDIONE 3-OXIME

$C_{12}H_{14}N_2O_2$  (II)

V. BERTOLASI, G. GILLI and A.C. VERONESE, 1982. Acta Cryst., B38, 502-511.

I. Monoclinic,  $B2_1/c$ ,  $a = 19.539$ ,  $b = 19.673$ ,  $c = 11.242$  Å,  $\beta = 92.80^\circ$ ,  $Z = 16$ . Cu radiation,  $R = 0.045$  for 2610 reflexions.

II. Orthorhombic,  $P2_12_12_1$ ,  $a = 7.296$ ,  $b = 14.747$ ,  $c = 11.177(3)$  Å,  $Z = 4$ . Cu radiation,  $R = 0.048$  for 1004 reflexions.

I and II (Fig. 1) have opposite configurations at the two centres of syn/anti isomerism related to the presence of the  $N(1)=C(8)$  and  $N(2)=C(10)$  bonds; I is the syn-methyl, syn-methyl and II the anti-methyl, anti-methyl isomer. The  $N(1)-C(8)-C(10)-N(2)$  torsion angles are  $-177.6(2)$  and  $-167.0(2)^\circ$  in I and  $-104.8(3)^\circ$  in II reflecting the fact that this group of atoms is approximately planar in I but not in II. The phenyl ring is approximately perpendicular to the  $C(1), N(1), C(8)$  plane in both compounds. Some bond lengths are:  $C(8)-C(10)$  1.479(3) (average) in I and 1.493(5) Å in II,  $N(2)-O(1)$  1.390(2) and 1.389(2) Å in I and 1.386(4) Å in II,  $C(8)-N(1)$  1.274(4) and 1.283(2) in I and 1.288(2) Å in II. In both structures the molecules are linked into infinite, linear chains by  $O(1)-H \dots N(1)$  hydrogen bonds, I having two parallel systems of such chains.

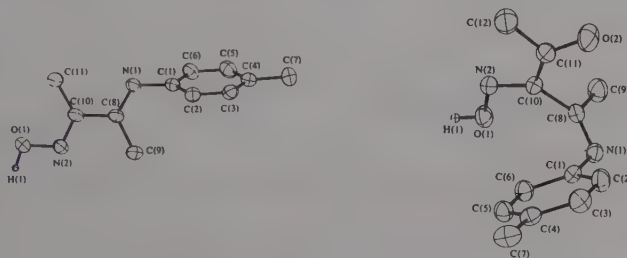


Fig. 1. The  $C_{11}H_{14}N_2O$  and  $C_{12}H_{14}N_2O_2$  molecules.

2-HYDROXYIMINOMETHYL-4'-CARBAMOYL-1,1'-OXY BIS(METHYLENE)DIPYRIDINIUM DICHLORIDE HYDRATE

$C_{14}H_{16}Cl_2N_4O_3 \cdot H_2O$

W. VAN HAVERE, A.T.H. LENSTRA, H.J. GEISE, G.R. VAN DEN BERG and H.P. BENSCHOP, 1982. Bull. Soc. Chim. Belg., 91, 219-225.

Monoclinic,  $P2_1/n$ ,  $a = 13.347$ ,  $b = 8.988$ ,  $c = 15.191$  Å,  $\beta = 106.14^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.0829$  for 2018 reflexions.

The molecule has the E (syn) configuration. Bond lengths are consistent with contributions from canonical forms shown in Fig. 1. The oxime  $N-O$  and  $N=C$  distances are 1.390(2) and 1.286(3) Å respectively and the  $O-N-C$  angle is  $112.3(2)^\circ$ .

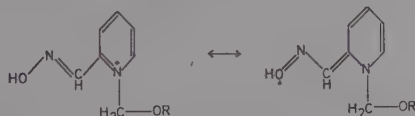


Fig. 1. Possible canonical forms for the oxime.

## AMMONIUM METHANETHIOSULPHONATE



G.K. COOPER, D.P. BLOXHAM and M. WEBSTER, 1982. J. Chem. Research, S, 104; M, 1166-1189.

Monoclinic,  $P2_1/c$ ,  $a = 8.832$ ,  $b = 8.117$ ,  $c = 7.697$  Å,  $\beta = 90.72^\circ$ ,  $D_m = 1.53$ ,  $Z = 4$ . Mo radiation,  $R = 0.034$  for 2042 reflexions.

The anion (Fig. 1) has approximate  $C_s$  symmetry with  $S(1)-S(2)$  1.954(1),  $S(2)-O$  1.466(1), 1.470(1) and  $S(2)-C$  1.767(1) Å. The angles at  $S(2)$  are within  $3^\circ$  of the tetrahedral angles. All the H atoms were located.

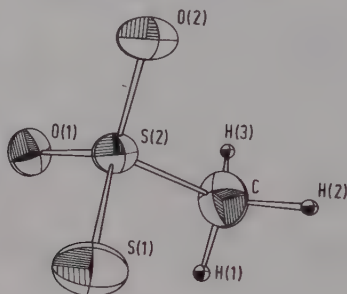
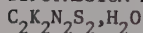


Fig. 1.  $\text{CH}_7\text{NO}_2\text{S}_2$ : the anion.

## DIPOTASSIUM N-CYANODITHIOCARBAMATE MONOHYDRATE



H. HLAWATSCHEK, M. DRAGER and G. GATTOW, 1982. Z. Anorg. Chem., 491, 145-153.

Orthorhombic,  $Pnma$ ,  $a = 10.336$ ,  $b = 7.862$ ,  $c = 9.882$  Å,  $D_m = 1.75(2)$ ,  $Z = 4$ . Mo radiation,  $R = 0.045$  for 1904 reflexions.

The structure (Fig. 1) comprises  $\text{K}^+$  cations and  $\text{S}_2\text{C}=\text{N}-\text{CN}^{2-}$  anions with  $m$  symmetry. The  $\text{K}^+$  is eight-coordinate (4 S, 3 N, O) with square-antiprism geometry (K-S 3.329(4), 3.307(4), 3.218(4), 3.380(4); K-N 2.939(9), 2.949(9), 3.803(9); K-O 2.750(9) Å). Bond lengths in the anion are: S-C(1) (mean) 1.723(1), C(1)-N(1) 1.342(2), N(1)-C(2) 1.305(2), C(2)-N(2) 1.164(2) Å ( $\text{S}_2-\text{C}(1)-\text{N}(1)-\text{C}(2)-\text{N}(2)$ ).

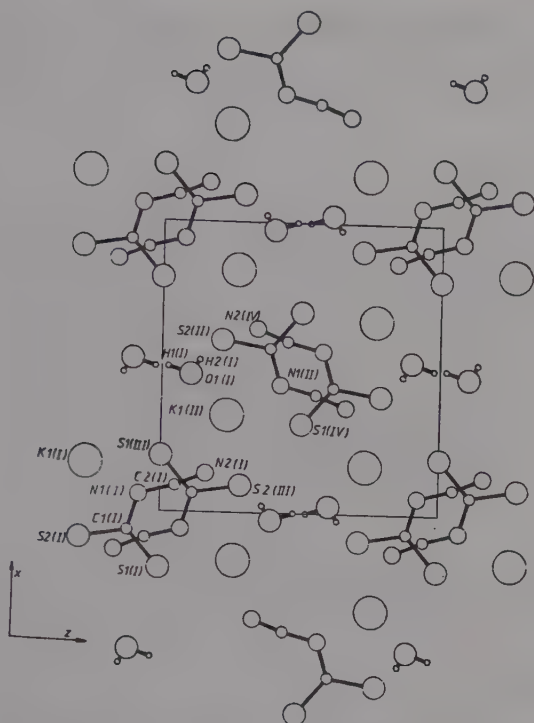
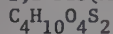


Fig. 1. Projection of the structure of  $2K^+(S_2C=N-CN)^{2-}, H_2O$  onto the  $x,z$ -plane.

1,2-BIS(METHYLSULFONYL)ETHANE



F. MO and Ø. BERG, 1982. Acta Chem. Scand., **A36**, 657-664.

Monoclinic,  $P2_1/c$ ,  $a = 6.260$ ,  $b = 5.949$ ,  $c = 10.643$  Å,  $\beta = 90.80^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.034$  for 2386 reflexions.

The molecule has crystallographic inversion symmetry (Fig. 1) and the sequence of bonds C-C-S-C(Me) is oriented gauche. Bond lengths are: S-O(1) 1.446, S-O(2) 1.443, S-C(1) 1.758, S-C(2) 1.779 and C(2)-C(2') 1.515 Å. Corresponding bond angles are: C(2)-S-O(1) 107.2, C(2)-S-O(2) 108.8, C(1)-S-O(1) 109.0, C(1)-S-O(2) 109.4, O(1)-S-O(2) 117.0, C(1)-S-C(2) 104.7, S-C(2)-C(2') 111.7.

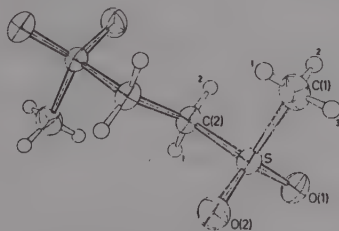
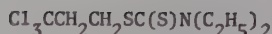
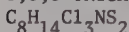


Fig. 1. Molecular structure of  $C_4H_{10}O_4S_2$ .



## 3,3,3-TRICHLOROPROPYL N,N-DIETHYLDITHIOCARBAMATE

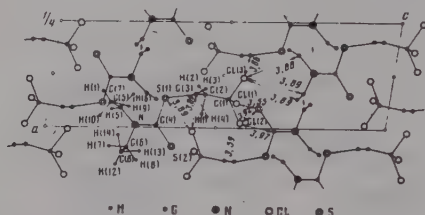


N.Kh. DZHAFAROV, M.K. GUSEINOVA, N.P. MUSTAFAEV and S.M. GUSEINZADE, 1982.

Kristallografiya, 27, 255-257 [Sov. Phys. Crystallogr., 27, 156-157].

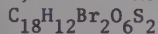
Monoclinic,  $P2_1/n$ ,  $a = 6.573$ ,  $b = 9.747$ ,  $c = 20.543$  Å,  $\beta = 97.99^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.058$  for 1388 reflexions.

The molecule (Fig. 1) consists of two planar fragments, containing atoms S(1), S(2), C(4), N, C(5), C(6) and S(1), C(1), C(2), C(3). The angle between these two planes is  $75^\circ$ . The planarity of the first group, and the bond lengths of N-C(4) 1.300 Å and C(4)-S(2) 1.677 Å intermediate between those characteristic of single and double bonds, indicate contributions from different resonance forms.



complex (II) (Fig. 1), the Cr atom has octahedral coordination, with Cr-S 2.368, 2.388(2), Cr-C 1.834-1.893(7) Å. The five-membered chelate ring Cr,S1,S2,C1,C2 is envelope shaped with a 13.3° angle at the Cr flap. The S-Cr-S angle is 84.55(6)°.

#### 2,4-HEXADIYNYLENE BIS(p-BROMOBENZENESULPHONATE)



R.L. WILLIAMS, D.J. ANDO, D. BLOOR, M. MOTEVALLI and M.B. HURSTHOUSE, 1982. *Acta Cryst.*, B38, 2078-2080.

Triclinic,  $P\bar{1}$ ,  $a = 9.066$ ,  $b = 5.028$ ,  $c = 11.175$  Å,  $\alpha = 80.91$ ,  $\beta = 94.30$ ,  $\gamma = 88.21^\circ$ ,  $D_m = 1.79$ ,  $Z = 1$ . Cu radiation,  $R = 0.039$  for 1376 reflexions.

The structure is very similar to that of the analagous, inactive, chloro-compound (1). The molecule (Fig. 1) lies on a centre of symmetry with C(1)-C(1') 1.392(10), C(1)-C(2) 1.175(7) Å and C(1')-C(1)-C(2) 177.7(6) and C(1)-C(2)-C(3) 178.2(6)°. The angle between the diacetylene units and the stacking direction (b-axis) is 65.6° which is outside the reactivity range for similar compounds of 40-49°.

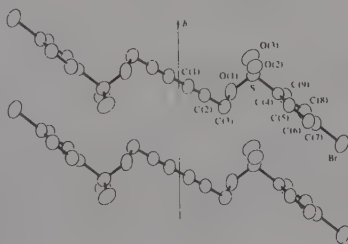
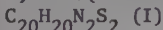


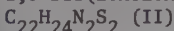
Fig. 1. The  $\text{C}_{18}\text{H}_{12}\text{Br}_2\text{O}_6\text{S}_2$  structure perpendicular to the ab plane.

#### 1. Structure Reports, 44B, 42.

#### 2,3-BIS(ETHYLTHIO)-2,3-DIPHENYL-1,4-BUTANEDINITRILE



#### 2,3-BIS(ETHYLTHIO)-2,3-BIS(p-TOLYL)-1,4-BUTANEDINITRILE



B. TINANT, E. ARTE, A. PARFONRY, J.P. DECLERCQ, G. GERMAIN and M. VAN MEERSSCHE, 1982. *Bull. Soc. Chim. Belg.*, 91, 621-628.

I. Triclinic,  $P\bar{1}$ ,  $a = 15.703$ ,  $b = 14.902$ ,  $c = 8.655$  Å,  $\alpha = 97.52$ ,  $\beta = 89.87$ ,  $\gamma = 75.59^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.052$  for 2845 reflexions.

II. Monoclinic,  $P2_1/n$ ,  $a = 16.872$ ,  $b = 12.082$ ,  $c = 10.329$  Å,  $\beta = 94.16^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.040$  for 1381 reflexions.

There are two independent molecules in I, and in both I and II (Fig. 1) the central C-C bond (C1-C13) is significantly elongated (1.597 and 1.603(7) in (I), 1.592(6) Å in II).

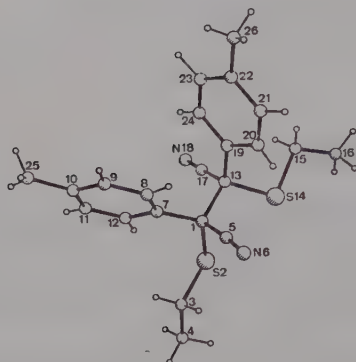
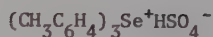


Fig. 1. A view of II,  $C_{22}H_{24}N_2S_2$ .

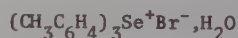
TRIS(p-TOLYL)SELENONIUM HYDROGEN SULPHATE  
 $C_{21}H_{22}O_4S_2$  (I)



TRIS(p-TOLYL)SELENONIUM CHLORIDE MONOHYDRATE  
 $C_{21}H_{21}ClSe, H_2O$  (II)



TRIS(p-TOLYL)SELENONIUM BROMIDE MONOHYDRATE  
 $C_{21}H_{21}BrSe, H_2O$  (III)



V.K. BEL'SKII, N.P. BEL'SKAYA, L.V. KONYAKHINA and V.P. SYSKOVA, 1982.

Kristallografiya, 27, 1102-1107 [Sov. Phys. Crystallogr., 27, 660-663].

I. Monoclinic,  $P2_1/b$ ,  $a = 11.088$ ,  $b = 11.028$ ,  $c = 19.016$  Å,  $\gamma = 119.17^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.039$  for 2202 reflexions.

II. Monoclinic,  $P2_1/n$ ,  $a = 10.625$ ,  $b = 10.844$ ,  $c = 18.579$  Å,  $\beta = 112.70^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.035$  for 1799 reflexions.

III. Monoclinic,  $P2_1/n$ ,  $a = 10.699$ ,  $b = 10.967$ ,  $c = 18.656$  Å,  $\beta = 114.09^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.049$  for 1657 reflexions.

In all three structures, the tris(p-tolyl)selenonium cation has a trigonal-pyramidal geometry (Fig. 1). The mean Se-C distance and the C-Se-C angles are 1.933 Å and  $100.8^\circ$  respectively. In I, the anions form centrosymmetric dimers (Fig. 1) by way of O-H...O hydrogen bonds (O...O 2.58 Å); one of the O atoms enters the coordination sphere of the Se atom thus increasing its coordination from trigonal pyramidal to distorted tetrahedral. The S-O distances range from 1.348 to 1.511 Å and the O-S-O angles from  $103.1$  to  $117.0^\circ$ . The Se...O distance is 3.056 Å. In II and III, dimerization of the hydrate occurs by way of O-H...Cl(Br) hydrogen bonds (Fig. 1) (O...Cl 3.175, 3.129 and O...Br 3.350, 3.397 Å). The Se coordination alters to distorted tetrahedral again because of Se...Cl (3.496 Å) and Se...Br (3.630 Å) bonds.

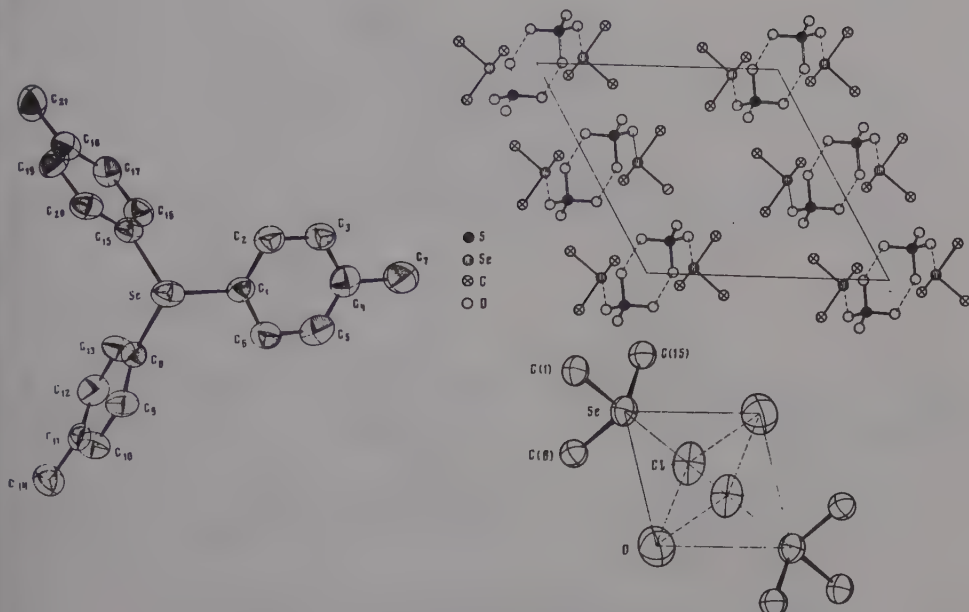


Fig. 1. General appearance of tri-p-selenonium cation (left), projection of  $C_{21}H_{22}O_4SSe$  on the (001) plane (top right), and dimers in  $C_{21}H_{21}ClSe \cdot H_2O$  and  $C_{21}H_{21}BrSe \cdot H_2O$  (bottom right).

(DIPHENYLMETHYLENE)AMINODIPHENYLSULPHONIUM HEXACHLOROANTIMONY METHYLENE CHLORIDE SOLVATE

$C_{25}H_{20}Cl_6N_2Sb, CH_2Cl_2$  (I)

$C_{25}H_{20}NS^+SbCl_6^-, CH_2Cl_2$

((DIPHENYLMETHYLENE)AMINO)TRIPHENYLPHOSPHONIUM HEXACHLOROANTIMONY METHYLENE CHLORIDE SOLVATE

$C_{31}H_{25}Cl_6NP_3Sb, CH_2Cl_2$  (II)

$C_{31}H_{25}NP^+SbCl_6^-, CH_2Cl_2$

(DIPHENYLSULPHIMIDO)TRIPHENYLPHOSPHONIUM HEXACHLOROANTIMONY METHYLENE CHLORIDE SOLVATE

$C_{30}H_{25}Cl_6NPSSb, CH_2Cl_2$  (III)

$C_{30}H_{25}NPS^+SbCl_6^-, CH_2Cl_2$

R. RECK, L. ZSOLNAI, G. HUTTNER, S. HERZBERGER and J.C. JOCHIMS, 1982. Chem. Ber., 115, 2981-2996.

I. Monoclinic,  $P2_1/c$ ,  $a = 15.31$ ,  $b = 17.06$ ,  $c = 15.78 \text{ \AA}$ ,  $\beta = 129.85^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.056$  for 2026 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 10.75$ ,  $b = 12.58$ ,  $c = 13.46 \text{ \AA}$ ,  $\alpha = 104.7$ ,  $\beta = 100.7$ ,  $\gamma = 102.6^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.052$  for 3128 reflexions.

III. Monoclinic,  $P2_1/c$ ,  $a = 13.96$ ,  $b = 13.92$ ,  $c = 20.33 \text{ \AA}$ ,  $\beta = 108.03^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.065$  for 2320 reflexions.

The structures of the cations showing the major bond lengths are displayed in Fig. 1.

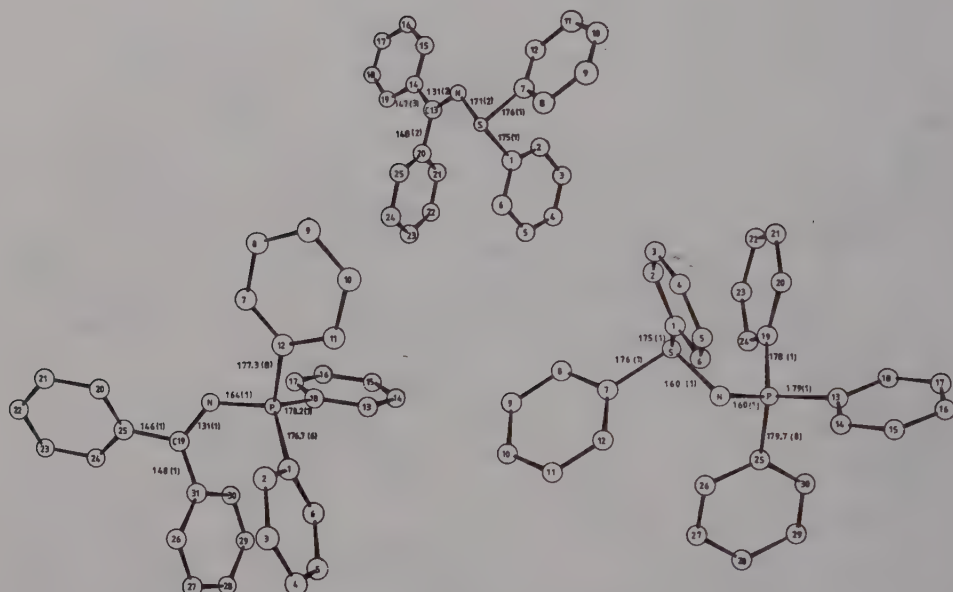


Fig. 1. The structures of  $C_{25}H_{20}NS^+$  (top),  $C_{31}H_{25}NP^+$  (lower left) and  $C_{30}H_{25}NPS^+$  (lower right), with major bond lengths (pm).

# p-CHLORO BENZOIC ACID

$C_7H_5ClO_2$

M. COLAPIETRO and A. DOMENICANO, 1982. Acta Cryst., B38, 1953-1957.

Triclinic,  $P\bar{1}$ ,  $a = 14.376$ ,  $b = 6.213$ ,  $c = 3.854$  Å,  $\alpha = 88.66$ ,  $\beta = 100.23$ ,  $\gamma = 93.23^\circ$ ,  $D_m = 1.535$ ,  $Z = 2$ . Mo radiation,  $R = 0.036$  for 1431 reflexions.

This refinement of the previously determined structure (1) yields the molecular dimensions shown (Fig. 1). The ring has  $C_{2v}$  ( $mm$ ) symmetry within experimental error. Cl and C(7) are displaced on the same side of the ring by 0.023 and 0.011 Å respectively. The C(7), O(1), O(2) plane makes an angle of  $5.7^\circ$  with the ring plane. Molecules are hydrogen bonded in pairs across a centre of symmetry ( $0 \dots 0.2618(2)$  Å).

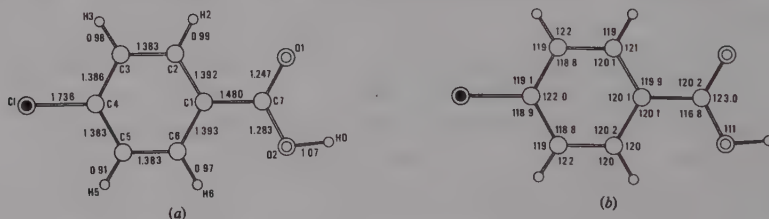


Fig. 1. Bond lengths and angles in p-chlorobenzoic acid. (e.s.d.s. involving non-H atoms are 0.002-0.003 Å and  $0.13$ - $0.18^\circ$ .)

1. Structure Reports, 40B, 102.

p-IODOBENZOIC ACID  
 $C_7H_5IO_2$

Y. TAKAKI, A. KURISI and K. NAKATA, 1982. Bull. Chem. Soc. Jpn., 55, 319-320.

Monoclinic,  $P2_1/a$ ,  $a = 30.36$ ,  $b = 6.04$ ,  $c = 4.16$  Å,  $\beta = 97.4^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.168$  for powder data.

The crystal structure was solved via X-ray powder data, packing analysis and pattern fitting. The structure consists of centrosymmetric hydrogen-bonded dimers ( $O \cdots O$  2.65 Å).

o-FLUOROBENZAMIDE  
 $C_7H_6FNO$

Y. KATO and K. SAKURAI, 1982. Bull. Chem. Soc. Jpn., 55, 1643-1644.

Monoclinic,  $P2_1/a$ ,  $a = 12.695$ ,  $b = 20.534$ ,  $c = 5.133$  Å,  $\beta = 97.80^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.076$  for 1471 reflexions.

There are two independent molecules in the asymmetric unit connected by one pair of hydrogen bonds to form asymmetric dimers (Fig. 1). These dimers are further linked by N-H...O hydrogen bonds into endless chains along the c-axis ( $N \cdots O$  2.917-2.945 Å).

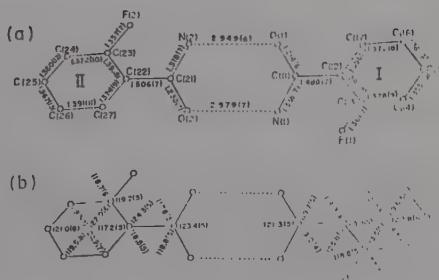


Fig. 1. The o-fluorobenzamide molecules and dimensions.

3,5-DIAMINO-2,4,6-TRINITROBENZAMIDE  
 $C_7H_6N_6O_7$

H.L. AMMON and S.K. BHATTACHARJEE, 1982. Acta Cryst., B38, 2083-2086.

Monoclinic,  $P2_1/n$ ,  $a = 16.557$ ,  $b = 10.2453$ ,  $c = 5.9400$  Å,  $\beta = 91.840^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.036$  for 1645 reflexions.

The benzene ring is slightly folded along  $C(2) \cdots C(5)$  (Fig. 1) with substituents deviating substantially (up to 0.297 Å) from the ring plane; the maximum and average deviations of the six ring atoms from the mean plane are 0.090 and 0.057 Å. The amide group is twisted  $70^\circ$  out-of-plane and the nitro group torsion angles are  $4.5(3)$ ,  $17.5(3)$  and  $34.8(2)^\circ$ . The average of four ring bond lengths at  $NH_2$  is 1.434(3) Å and average internal C-C-C angles at  $NH_2$  and  $NO_2$  are  $116.2(2)$  and  $122.2(2)^\circ$ . There are strong intramolecular and weak intermolecular N-H...O bonds with molecules linked into sheets almost normal to  $b$ .



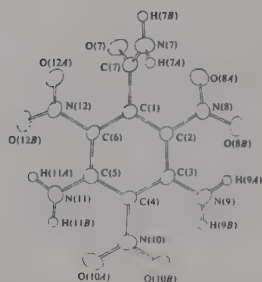


Fig. 1. The 3,5-diamino-2,4,6-trinitrobenzamide molecule.

m-HYDROXYBENZOIC ACID (CENTROSYMMETRIC POLYMORPH)

$C_7H_6O_3$  (I)

$HOC_6H_4COOH$

m-HYDROXYBENZOIC ACID (NON-CENTROSYMMETRIC POLYMORPH)

$C_7H_6O_3$  (II)

$HOC_6H_4COOH$

G.V. GRIDUNOVA, N.G. FURMANOVA, Yu.T. STRUCHKOV, Z.I. EZHKOVA, L.P. GRIGOR'EVA and B.A. CHAYANOV, 1982. Kristallografiya, 27, 267-272 [Sov. Phys. Crystallogr., 27, 164-167].

I. Monoclinic,  $P2_1/b$ ,  $a = 5.493$ ,  $b = 23.893$ ,  $c = 4.943$  Å,  $\gamma = 105.70^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.055$  for 1047 reflexions.

II. Orthorhombic,  $Pna2_1$ ,  $a = 20.075$ ,  $b = 3.751$ ,  $c = 8.291$  Å,  $Z = 4$ . Cu radiation,  $R = 0.047$  for 361 reflexions.

Bond lengths in the two molecules are shown in Fig. 1. The interesting feature is that in I the lengths of C(7)-O(1) and C(7)-O(2) are equivalent whilst in II they are decidedly non-equivalent at 1.324 and 1.209 Å. The crystal structure of I consists of layers of centrosymmetric dimers (O-H...O 2.618 Å between carboxyl groups) with layers linked by hydrogen bonds between OH groups (O-H...O 2.796 Å). The molecules of II are linked in infinite chains with hydrogen bonds between carboxyl and hydroxy groups (O-H...O 2.683 and 2.725 Å).

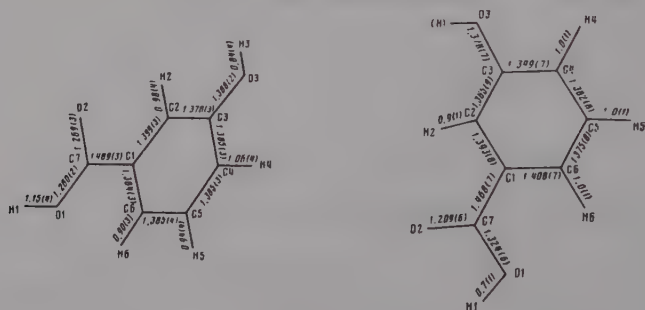


Fig. 1. Bond lengths in  $C_7H_6O_3$  (I) (left) and (II) (right).

IODOPHENACYL

$C_8H_7IO$

$C_6H_5COCH_2I$

J.-P. LERE-PORTE, A. BONNIOL, J. PETRISSANS and M.-C. BRIANSO, 1982. Acta Cryst., B38, 1035-1037.

Monoclinic,  $P2_1/c$ ,  $a = 8.560$ ,  $b = 10.803$ ,  $c = 8.889$  Å,  $\beta = 99.84^\circ$ ,  $Z = 4$ . Radiation not given,  $R = 0.04$  for 1131 reflexions (at 193 K).

The molecule (Fig. 1) exhibits a gauche conformation in which the dihedral angle between the C=O and C-I bonds is  $93^\circ$ . The shortest intermolecular I...I and I...O contacts are respectively 4.02 and 3.24 Å.

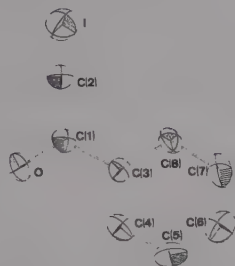


Fig. 1. A view of the iodophenacyl molecule.

# 2-HYDROXY-5-METHYLBENZOIC ACID

$C_8H_8O_3$  (I)

# [2(o)]-2,5-DIHYDROXYBENZOIC ACID

$C_7H_6O_4$  (II)

# [2(d)]-2,5-DIHYDROXYBENZOIC ACID

$C_7H_6O_4$  (III)

M. HAISA, S. KASHINO, S.-I. HANADA, K. TANAKA, S. OKAZAKI and M. SHIBAGAKI, 1982. Acta Cryst., B38, 1480-1485.

I. Monoclinic,  $P2_1/a$ ,  $a = 21.24$ ,  $b = 5.114$ ,  $c = 3.307$  Å,  $\beta = 103.76^\circ$ ,  $D_m = 1.31$ ,  $Z = 4$ . Cu radiation,  $R = 0.073$  for 904 reflexions.

II. Monoclinic,  $P2_1/a$ ,  $a = 23.945$ ,  $b = 4.908$ ,  $c = 5.621$  Å,  $\beta = 100.981^\circ$ ,  $D_m = 1.57$ ,  $Z = 4$ . Cu radiation,  $R = 0.035$  for 961 reflexions.

III. Monoclinic,  $Pa$ ,  $a = 11.229$ ,  $b = 11.830$ ,  $c = 4.966$  Å,  $\beta = 90.50^\circ$ ,  $D_m = 1.55$ ,  $Z = 4$ . Cu radiation,  $R = 0.039$  for 960 reflexions.

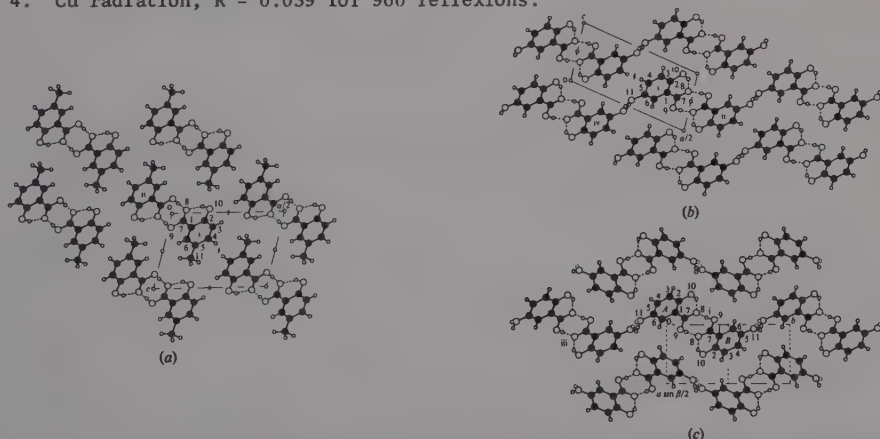


Fig. 1. The crystal structures of (a) 2-hydroxy-5-methylbenzoic acid and (b) and (c) the two dimorphs of 2,5-dihydroxy benzoic acid. Broken lines show hydrogen bonds and in (c) the disordered H atoms are shown.



The molecules are linked in the crystal (Fig. 1) in infinite interlocking hydrogen-bonded ribbons extending parallel to *b* (O-H...O 2.673 Å) by the action of the corresponding space-group screw axis. The plane of the carboxy group is inclined to that of the phenyl ring by 56.2°. The C(2) and C(6) methoxy groups make angles of 21.6 and 2.8° respectively, with the phenyl plane.

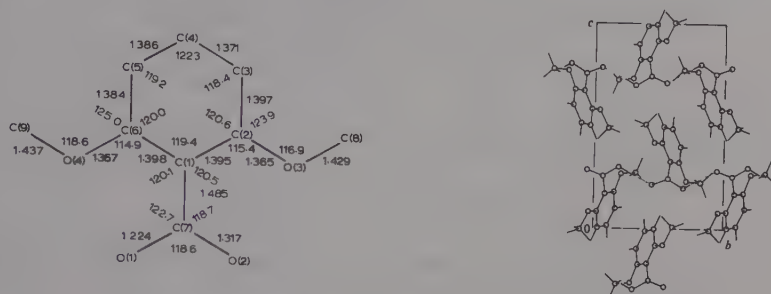
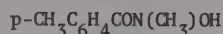


Fig. 1. 2,6-dimethoxybenzoic acid: bond lengths and angles (left) and the molecular packing seen in *a*-axis projection (right).

# N-p-TOLUYL-N-METHYLHYDROXYLAMINE



V.N. KALININ, M.Yu. ANTIPIN, V.M. YURCHENKO and Yu.T. STRUCHKOV, 1982. Zh. Strukt. Khim., 23-5, 83-87 [J. Struct. Chem., 23, 723-727].

Monoclinic,  $P2_1/n$ ,  $a = 12.106$ ,  $b = 10.198$ ,  $c = 7.202$  Å,  $\beta = 99.25^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.058$  for 1174 reflexions.

The molecule (Fig. 1) has a *trans* conformation, stabilized by an intermolecular O-H...O hydrogen bond (O...O 2.636(2) Å). The aromatic ring is rotated 48° relative to the  $-(\text{O})\text{CN}(\text{CH}_3)\text{O}$  plane. The N atom has flattened pyramidal coordination.

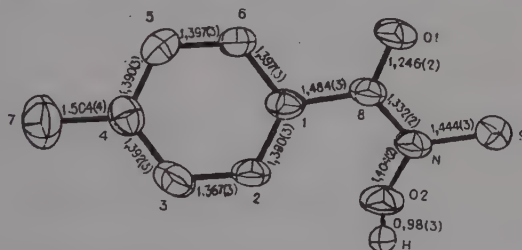
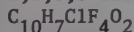


Fig. 1. The  $\text{CH}_3\text{C}_6\text{H}_4\text{CON}(\text{CH}_3)\text{OH}$  molecule.

# 2,2,3,3-TETRAFLUOROPROPYL-p-CHLORO BENZOATE



R. TOUILLAX, G. GERMAIN, J.P. DECLERCQ, M. VAN MEERSSCHE, C. WILANTE and G. LEROY, 1982. J. Fluorine Chem., 20, 3-8.

Monoclinic,  $P2_1$ ,  $a = 4.826$ ,  $b = 9.036$ ,  $c = 12.442$  Å,  $\beta = 94.64^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.032$  for 728 reflexions.

The molecular structure may be considered as three linked planar units (Fig. 1). The hydrocarbon chain O(10), C(11), C(12), C(15) and F(17) is practically planar as is the carbonyl grouping (O(9), C(8), O(10), C(11)). The dihedral angle between these two planes is 100.9°, and the angle between the carbonyl grouping and the

phenyl ring plane is  $8^\circ$ . Some bond lengths of interest are: C1(1)-C(2) 1.740, C(5)-C(8) 1.476, C(8)-O(9) 1.203, C(8)-O(10) 1.345, O(10)-C(11) 1.441, C(11)-C(12) 1.492, C(12)-F(13) 1.348, C(12)-F(14) 1.370, C(12)-C(15) 1.511, C(15)-F(16) 1.348, C(15)-F(17) 1.360, C(15)-H(C15) 0.980 Å.

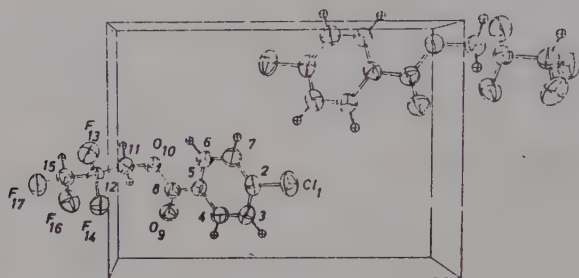


Fig. 1. View of the unit cell of  $C_{10}H_7ClF_4O_2$ .

#### 2-ACETOXY-6-METHYLBENZOIC ACID



F.R. FRONCZEK, M.L. MERRILL and R.D. GANDOUR, 1982. *Acta Cryst.*, B38, 1337-1339.

Monoclinic,  $P2_1/n$ ,  $a = 10.164$ ,  $b = 7.238$ ,  $c = 13.186$  Å,  $\beta = 96.74^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.041$  for 1260 reflexions.

In the crystal the molecule (Fig. 1) forms centrosymmetric dimers with O-H...O hydrogen bonds of length 2.621 Å. The carboxyl group is rotated  $44.7^\circ$  from the plane of the benzene ring; this rotation is significant when compared to aspirin, and is likely to be caused by steric interactions of the methyl group juxtaposed to the carbonyl.

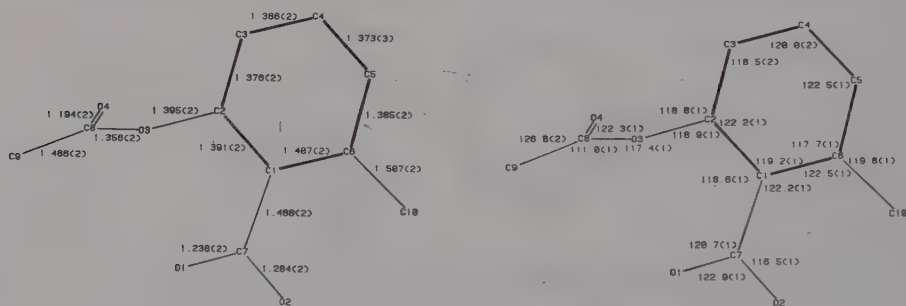


Fig. 1.  $C_{10}H_{10}O_4$ : bond distances (left) and angles (right).

#### 4-t-BUTYLBENZOIC ACID



H. VAN KONINGSVELD, 1982. *Cryst. Struct. Comm.*, 11, 1423-1433.

Triclinic,  $P\bar{1}$ ,  $a = 11.068$ ,  $b = 13.385$ ,  $c = 13.625$  Å,  $\alpha = 100.64$ ,  $\beta = 90.91$ ,  $\gamma = 97.74^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.049$  for 4036 reflexions (at  $-160^\circ C$ ).

The crystal structure consists of chains of molecules approximately parallel to the [113] direction. Within each chain, the four independent molecules form two dimers through hydrogen bonding between carboxyl groups (O...O 2.614-2.691(4) Å).

The orientations of the t-butyl groups relative to their benzene rings is shown in Fig. 1

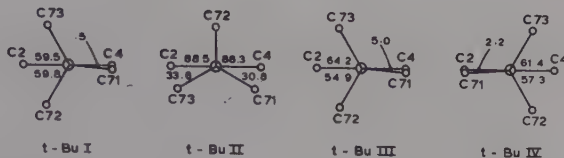


Fig. 1. Newman projections showing the orientations of the four independent t-butyl groups in the 4-t-butylbenzoic acid structure.

## DIETHYL TEREPHTHALATE



J. HASEK, V. LANGER and J. JECNY, 1982. *Acta Cryst.*, B38, 1662-1663.

Monoclinic,  $P2_1/n$ ,  $a = 9.140$ ,  $b = 15.450$ ,  $c = 4.225$ ,  $\beta = 92.89^\circ$ ,  $D_m = 1.243$ ,  $Z = 2$ .  
Cu radiation,  $R = 0.065$  for 922 reflexions.

In this redetermined structure (1) the C(4)-O(8) and C(6)-C(7) bond lengths (Fig. 1) are now in good agreement with expected values.

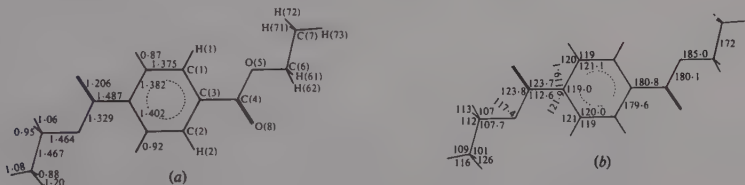
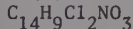


Fig. 1. (a) Bond lengths and (b) angles in diethyl terephthalate.

1. Structure Reports, 12, 384.

## 2-(2,6-DICHLOROPHENYLCARBAMOYL) BENZOIC ACID



C.H.L. KENNARD, G. SMITH and G.F. KATEKAR, 1982. *Aust. J. Chem.*, 35, 1933-1937.

Monoclinic,  $P2_1/c$ ,  $a = 12.150$ ,  $b = 13.629$ ,  $c = 8.368$  Å,  $\beta = 103.75^\circ$ ,  $D_m = 1.54$ ,  $Z = 4$ .  
Mo radiation,  $R = 0.076$  for 971 reflexions.

The carboxylic acid group A (Fig. 1) is approximately coplanar with ring B (C(11)C(12)-C(121)O(121) 2.78°) which in turn is nearly coplanar with ring C (dihedral angles are: A-B 7.63, B-C 4.14, A-C 11.6°). The amide group adopts a synclinal conformation (N(1)C(111)-C(11)C(12) 77.1°). In the crystal, molecules form centrosymmetric hydrogen-bonded dimers (O(122)H...O(121') 2.64(1)°).



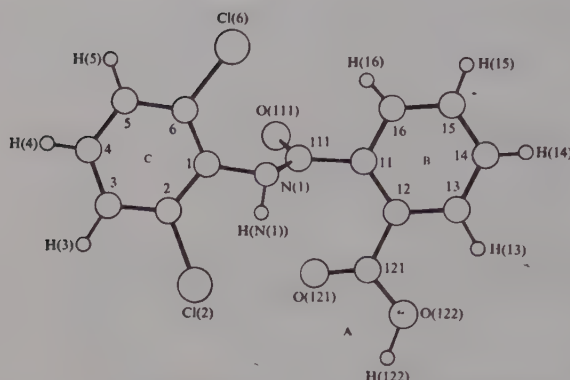


Fig. 1. The molecular conformation of 2-(2,6-dichlorophenylcarbamoyl)benzoic acid.

2-([3-(TRIFLUOROMETHYL)PHENYL]AMINO)BENZOIC ACID (FLUFENAMIC ACID)  
 $C_{14}H_{10}F_3NO_2$

H.M. KRISHNAMURTHY, T.N. BHAT and M. VIJAYAN, 1982. Acta Cryst., B38, 315-317.

Monoclinic,  $P2_1/c$ ,  $a = 12.523$ ,  $b = 7.868$ ,  $c = 12.874$  Å,  $\beta = 95.2^\circ$ ,  $D_m = 1.47$ ,  $Z = 4$ .  
 Mo radiation,  $R = 0.074$  for 2255 reflexions.

In the crystal structure (Fig. 1) pairs of centrosymmetric molecules are linked into dimers by hydrogen bonds with  $O \cdots O$  2.647 Å. The molecule also contains an intramolecular hydrogen bond with  $N \cdots O$  2.646 Å. The carboxyl group and the phenyl ring bearing the carboxyl group are nearly coplanar but the two phenyl rings are inclined at  $52.8^\circ$  with respect to each other. The polymorph of flufenamic acid presently studied differs from that previously studied (1) in the geometrical disposition of the [3-(trifluoromethyl)phenyl] amino moiety with respect to the benzoic acid moiety.

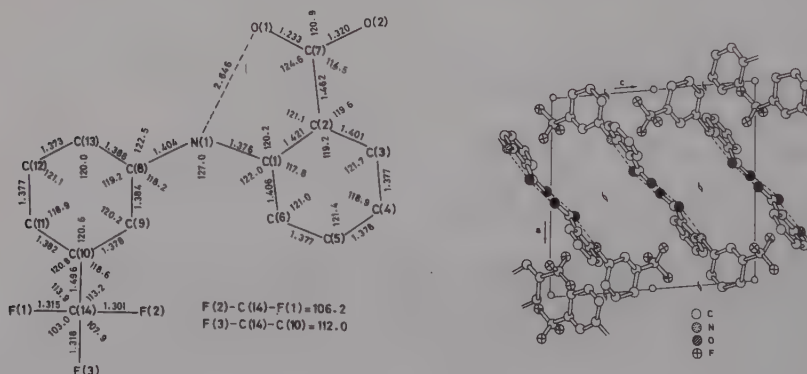
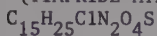


Fig. 1. Bond lengths and angles in  $C_{14}H_{10}F_3NO_2$ , and a view along the  $b$  axis of the crystal structure, in which dashed lines indicate hydrogen bonds.

1. Structure Reports, 39B, 66.

5-(METHYLSULPHONYL)-N-[(2-DIETHYLAMINO)ETHYL]-2-METHOXYBENZAMIDE HYDROCHLORIDE  
(TIAPRIDE HYDROCHLORIDE)



N.M. BLATON, O.M. PEETERS, C.J. DE RANTER, O. DENISOFF and L. MOLLE, 1982. Cryst. Struct. Comm., 11, 1357-1362.

Monoclinic,  $P2_1/a$ ,  $a = 8.192$ ,  $b = 22.393$ ,  $c = 10.124$  Å,  $\beta = 98.67^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.065$  for 1744 reflexions.

No unusual bond distances or angles are observed in the molecule. An intra-molecular hydrogen bond between N13-H13 and O7 stabilises the structure. The plane defined by the benzene ring makes a dihedral angle of  $17.0^\circ$  with that defined by the amide group.

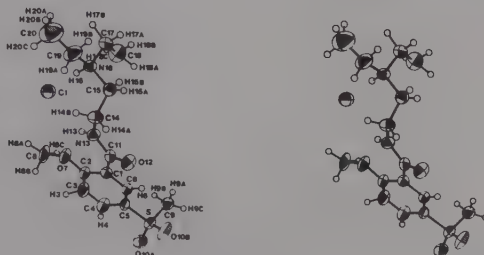


Fig. 1. A stereoview of  $C_{15}H_{25}ClN_2O_4S$ .

METHYL 3-(p-NITROPHENYL)-3-[(p-NITROBENZOYL)OXY]-2-CYANOPROPENOATE



M.J. STRAUSS and Z. RAPPOPORT, 1982. J. Org. Chem., 47, 4809-4812.

Monoclinic,  $P2_1/c$ ,  $a = 15.183$ ,  $b = 8.581$ ,  $c = 14.39$  Å,  $\beta = 102.53^\circ$ .

The X-ray analysis showed that the molecule is the Z-isomer. The C=C distance is  $1.34(1)$  Å (Fig. 1).

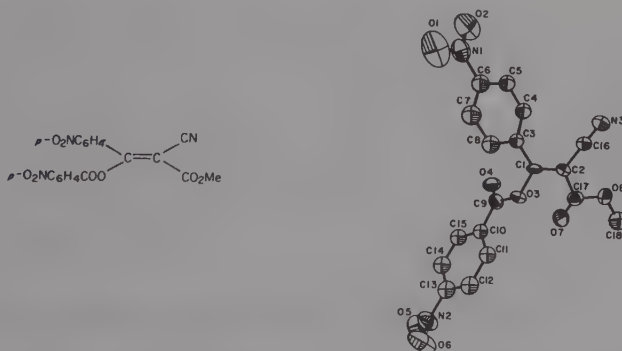


Fig. 1. A view of  $C_{18}H_{11}N_3O_8$ .

## 1,4-BUTANEDIYL DIBENZOATE



G. BOCELLI and M.-F. GRENIER-LOUSTALOT, 1982. *Acta Cryst.*, **B38**, 2072-2075.

Triclinic,  $\bar{P}1$ ,  $a = 12.591$ ,  $b = 8.555$ ,  $c = 7.854$  Å,  $\alpha = 115.3$ ,  $\beta = 90.2$ ,  $\gamma = 83.7^\circ$ ,  $[Z = 2]$ . Cu radiation,  $R = 0.057$  for 2725 reflexions.

A significant shortening is present in the  $\text{Csp}^3\text{-Csp}^3$  bonds of the glycol part of the molecule (Fig. 1) with  $\text{C}(8)\text{-C}(9)$  1.462(2) and  $\text{C}(10)\text{-C}(11)$  1.448(2) while  $\text{C}(9)\text{-C}(10)$  is 1.521(5) Å. The glycol fragment has the trans-trans-gauche conformation. The two carboxylic groups are inclined at  $6.3(3)^\circ$  and  $1.1(3)^\circ$  respectively to the benzene rings. The mean ring bonds are 1.363(6) and 1.359(8) Å and the two rings are inclined at  $58.5(4)^\circ$ .

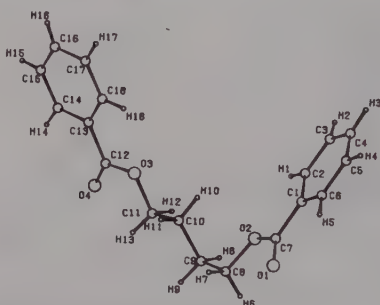
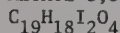


Fig. 1. The 1,4-butanediyl dibenzoate molecule.

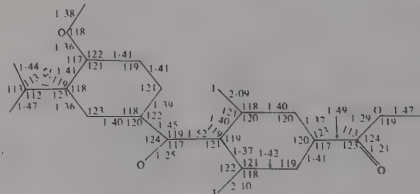
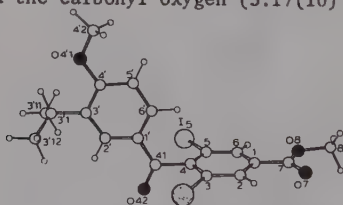
## METHYL 3,5-DIIDO-4-(3-ISOPROPYL-4-METHOXYBENZOYL) BENZOATE



V. CODY, E. CHEUNG and E.C. JORGENSEN, 1982. *Acta Cryst.*, **B38**, 2270-2272.

Orthorhombic,  $\text{Iba}2$ ,  $a = 20.998$ ,  $b = 24.002$ ,  $c = 8.032$  Å,  $Z = 8$ . Mo radiation,  $R = 0.066$  for 2281 reflexions.

In the molecule (Fig. 1) the conformation of the diphenyl ketone bridge is skewed and the 3'-isopropyl group distally oriented, as is observed for many thyroid hormone analogue structures. There is a short  $\text{I} \cdots \text{O}$  intermolecular contact between  $\text{I}(5)$  and the carbonyl oxygen (3.17(10) Å).



The two molecules in the asymmetric unit (Fig. 1) have a widely stretched, nearly linear form. They are packed in an approximate parallel imbricated mode, the molecular long axes making an angle of about  $5^\circ$  with the crystal *c* axis. The geometry and packing are discussed in relation to the mesomorphic behaviour of the molecule.

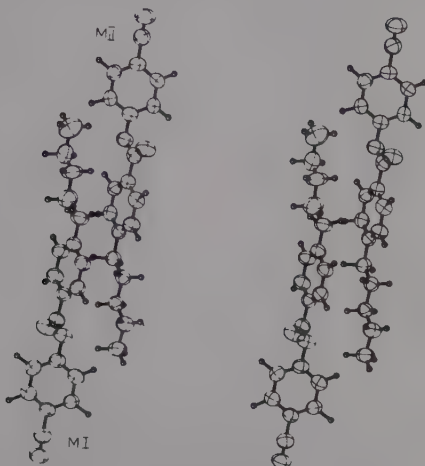
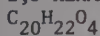


Fig. 1. A stereoview of the two  $C_{19}H_{19}NO_2$  molecules.

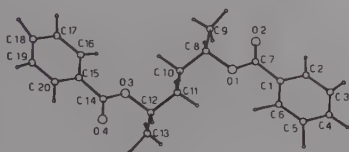
#### 2,5-HEXANEDIYL DIBENZOATE



G. BOCELLI and M.F. GRENIER-LOUSTALOT, 1982. *Acta Cryst.*, B38, 3135-3139.

Orthorhombic,  $P2_12_12_1$ ,  $a = 16.992$ ,  $b = 11.666$ ,  $c = 9.318 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.047$  for 1022 reflexions.

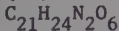
The two phenyl rings (Fig. 1) are tilted at  $9.1^\circ$  to each other. The 'tetramethylene' part of the chain presents the usual shortening of the  $-\text{CH}_2-\text{CH}_2-$  distances in two bonds only and it has gauche-trans-gauche conformation.



O(1)-C(7)	1.333 (6)	C(8)-C(9)	1.500 (7)
O(1)-C(8)	1.483 (6)	C(8)-C(10)	1.498 (7)
O(2)-C(7)	1.201 (6)	C(10)-C(11)	1.512 (6)
O(3)-C(12)	1.465 (6)	C(11)-C(12)	1.523 (7)
O(3)-C(14)	1.337 (8)	C(12)-C(13)	1.501 (6)
O(4)-C(14)	1.205 (6)	C(14)-C(15)	1.482 (9)
C(1)-C(2)	1.377 (5)	C(15)-C(16)	1.382 (6)
C(1)-C(6)	1.399 (6)	C(16)-C(20)	1.376 (7)
C(1)-C(7)	1.457 (6)	C(16)-C(17)	1.394 (7)
C(2)-C(3)	1.388 (6)	C(17)-C(18)	1.355 (7)
C(3)-C(4)	1.354 (7)	C(18)-C(19)	1.403 (8)
C(4)-C(5)	1.413 (6)	C(19)-C(20)	1.367 (7)
C(5)-C(6)	1.396 (7)		

Fig. 1. The  $C_{20}H_{22}O_4$  molecule and bond lengths.

#### (2R,4R-2S,4S)-5,5-DIMETHYL-4-PHENYL-2-HEXYL 3,5-DINITROBENZOATE



A. DE ANDRÉS, J.L. BALCAZAR and J. FAYOS, 1982. *Acta Cryst.*, B38, 2910-2911.

Monoclinic,  $P2_1/c$ ,  $a = 14.549$ ,  $b = 11.195$ ,  $c = 13.694 \text{ \AA}$ ,  $\beta = 110.15^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.059$  for 2700 reflexions.

In the molecule (Fig. 1) bond lengths and angles are generally as expected. The conformations at the two chiral centres, separated by a methylene bridge, are compared with those theoretically predicted for the free carbinol.

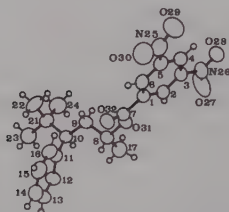
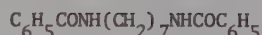
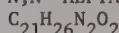


Fig. 1.  $C_{21}H_{24}N_2O_6$ : perspective view of the molecule.

**N,N'-HEPTAMETHYLENEDIBENZAMIDE**



J. BRISSON and F. BRISSE, 1982. *Acta Cryst.*, **B38**, 2663-2667.

Monoclinic,  $P2_1/n$ ,  $a = 5.087$ ,  $b = 44.172$ ,  $c = 8.496$  Å,  $\beta = 91.08^\circ$ ,  $D_m = 1.14$ ,  $Z = 4$ .  
Cu radiation,  $R_w = 0.054$  for 1501 reflexions.

The molecule (Fig. 1) consists of a central nearly planar  $NH-(CH_2)_7-NH$  sequence between two benzamide groups. The dihedral angles between the methylenic sequence and the benzene rings are  $117.2(18)$  and  $124.5(4)^\circ$  respectively. The distance between the centres of the benzene rings is  $16.789(8)$  Å. Each molecule is connected through four hydrogen bonds to two others, forming ribbons parallel to the  $ab$  plane and extending in the  $a$  direction. This hydrogen bonding is characterised by the following parameters:  $N(1)-H(N1) \dots O(1')$   $2.07(3)$  Å,  $N(1)-H(N1) \dots O(1')$   $151(3)^\circ$  and  $N(2)-H(N2) \dots O(2')$   $2.03(3)$  Å,  $N(2)-H(N2) \dots O(2')$   $153(3)^\circ$ .

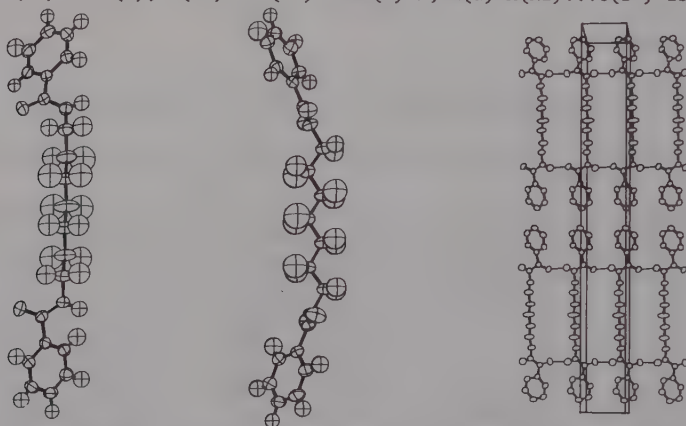
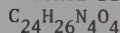


Fig. 1.  $C_{21}H_{26}N_2O_2$ : perspective views of the molecule (left) and of the crystal structure (right); hydrogen bonds are shown in the latter.

**DIETHYL BIS-(2-AMINOPHENYL)-p-DIAMINOTEREPTHALATE**



B.J. MANN, R.B. WILSON, D.Y. CURTIN and I.C. PAUL, 1982. *Cryst. Struct. Comm.*, **11**, 163-168.

Monoclinic,  $P2_1/c$ ,  $a = 12.948$ ,  $b = 5.433$ ,  $c = 18.062$  Å,  $\beta = 120.85^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.044$  for 1623 reflexions.

The molecule (Fig. 1) lies about a crystallographic inversion centre. Intramolecular  $N-H \dots O$  hydrogen bonds keep the ester groups effectively planar with the aromatic ring. The 2-aminophenyl groups are twisted about  $N(1)-C(7)$  through  $73^\circ$

from planarity. Molecules are linked in the crystal by additional N-H...O and N-H...N hydrogen bonds.

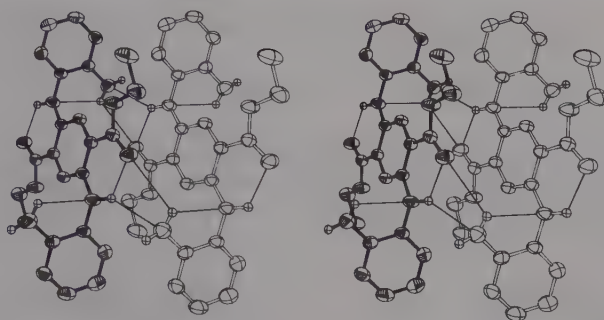


Fig. 1. A stereoview of a pair of  $C_{24}H_{26}N_4O_4$  molecules showing hydrogen bonding.

PHENYLGLYOXYLIC ACID 2-BENZOYLOXY-4,6-DI-*t*-BUTYLPHENYL ESTER

$C_{29}H_{30}O_5$

W. FRIEDRICHSEN, I. SCHWARZ and T. DEBAERDEMAEKER, 1982. Z. Naturforsch., 37b, 663-668.

Monoclinic,  $P2_1/a$ ,  $a = 19.416$ ,  $b = 9.766$ ,  $c = 13.757$  Å,  $\beta = 103.57^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.052$  for 1657 reflexions.

The structure was determined to be that shown in Fig. 1 with normal values for bond lengths and angles. The dihedral angle between planes C(110)-C(115) and C(1)-C(6) is  $80.9(5)^\circ$ .

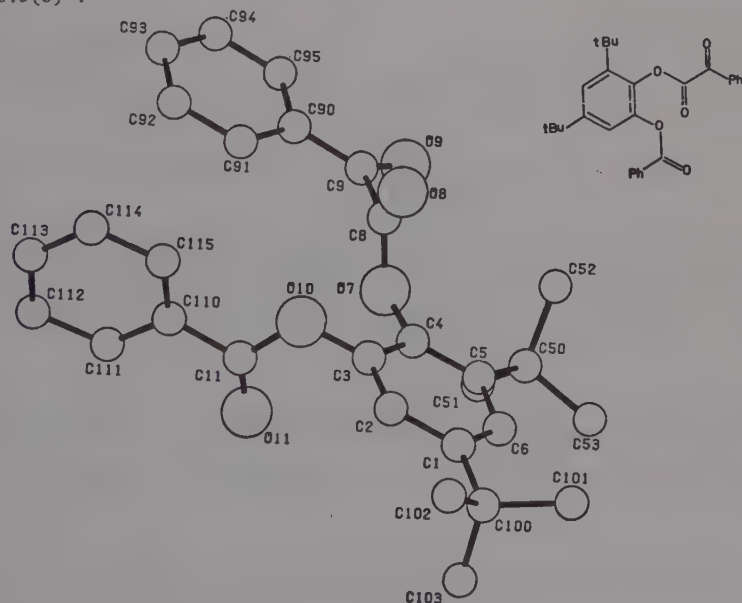
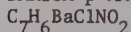


Fig. 1. The molecular structure of  $C_{29}H_{30}O_5$ .



## BARIUM p-AMINOBENZOATE CHLORIDE



I.R. AMIRASLANOV, F.N. MUSAEV and Kh.S. MAMEDOV, 1982. Zh. Strukt. Khim., 23-2, 118-120 [J. Struct. Chem., 23, 267-269].

Monoclinic,  $P2_1$ ,  $a = 10.460$ ,  $b = 6.944$ ,  $c = 5.821 \text{ \AA}$ ,  $\beta = 90.96^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.068$  for 940 reflexions.

The structure (Fig. 1) consists of layers of polymerized Ba polyhedra (perpendicular to the a-axis), linked to one another by bridging  $\text{H}_2\text{NC}_6\text{H}_4\text{COO}^-$  ligands. The Ba atoms are eight-coordinate: Ba-Cl 2.97(1), 3.13(1), 3.14(1), Ba-O 2.79(3), 3.00(3), 2.84(3), 2.93(3), Ba-N 2.85(4)  $\text{\AA}$ .

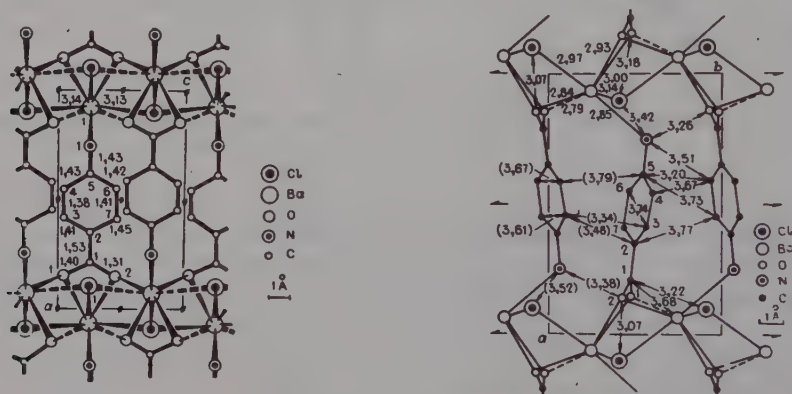
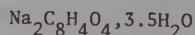
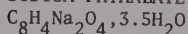


Fig. 1. Two views of the  $\text{Ba}(\text{H}_2\text{NC}_6\text{H}_4\text{COO})\text{Cl}$  structure.

## SODIUM PHTHALATE HYDRATE



T.B. TEPLOVA, T.A. SHIBANOVA, T.M. OKHRIMENKO and O.G. KARPINSKII, 1982. Kristallografiya, 27, 60-64 [Sov. Phys. Crystallogr., 27, 33-35].

Orthorhombic,  $Pbca$ ,  $a = 17.635$ ,  $b = 22.055$ ,  $c = 11.910 \text{ \AA}$ ,  $D_m = 1.566$ ,  $Z = 16$ . Mo radiation,  $R = 0.064$  for 744 reflexions.

The crystal structure consists of  $\text{Na}^+$  cations, phthalate anions and molecules of water of crystallization. The bond distances and valency angles in the two independent phthalate anions are shown in Fig. 1. The carboxyl C-O bond lengths in the "A" anion are all 1.26  $\text{\AA}$  whilst in the "B" anion they vary from 1.22 to 1.31  $\text{\AA}$ . Each  $\text{Na}^+$  ion is coordinated to six O atoms in distorted octahedral fashion, with  $\text{Na}^+\text{-O}$  distances ranging from 2.32 to 2.76  $\text{\AA}$ .

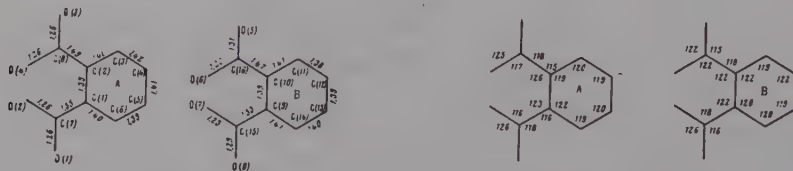


Fig. 1. Bond distances ( $\text{\AA}$ ) and valency angles ( $^\circ$ ) in the independent phthalate anions.

## POTASSIUM HYDROGEN BIS(p-FLUOROBENZOATE)



J. LONGO and M.F. RICHARDSON, 1982. *Acta Cryst.*, **B38**, 2482-2483.

Monoclinic,  $\text{C2/c}$ ,  $a = 32.337$ ,  $b = 3.817$ ,  $c = 11.239$  Å,  $\beta = 107.42^\circ$ ,  $D_m = 1.59$ ,  $Z = 4$ . Mo radiation,  $R = 0.052$  for 731 reflexions.

In the anion the phenyl ring is planar, with the carboxylate group twisted slightly out of the plane ( $8.9^\circ$ ) (Fig. 1). The acidic H atoms appear to be located on centres of symmetry, and link pairs of p-fluorobenzoate residues together by a strong symmetrical hydrogen bond with  $\text{O}\cdots\text{O}$  2.460(4) Å. The coordination of the  $\text{K}^+$  ion is a highly distorted octahedron with average  $\text{K}^+\cdots\text{O}$  distance 2.78(4) Å.

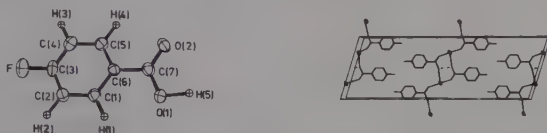
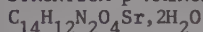


Fig. 1.  $\text{KH}(\text{p-FC}_6\text{H}_4\text{CO}_2)_2$ : views of the asymmetric unit of the centrosymmetric  $[\text{H}(\text{FC}_6\text{H}_4\text{CO}_2)_2]^-$  anion and of the unit cell contents; dashed lines indicate hydrogen bonds.

## STRONTIUM p-AMINOBENZOATE DIHYDRATE



I.R. AMIRASLANOV, F.N. MUSAIEV and Kh.S. MAMEDOV, 1982. *Zh. Strukt. Khim.*, **23-2**, 114-117 [*J. Struct. Chem.*, **23**, 263-266].

Orthorhombic,  $\text{P2}_12_12_1$ ,  $a = 6.86$ ,  $b = 13.39$ ,  $c = 18.58$  Å,  $Z = 4$ . Mo radiation,  $R = 0.086$  for 1353 reflexions.

The structure (Fig. 1) is based on infinite chains extending along the  $a$  axis. The eight-coordinate Sr polyhedra are linked by bridging oxygen atoms. The Sr-O distances range from 2.46(1) to 2.74(1) Å and the distance between neighboring Sr atoms is 4.07(1) Å.

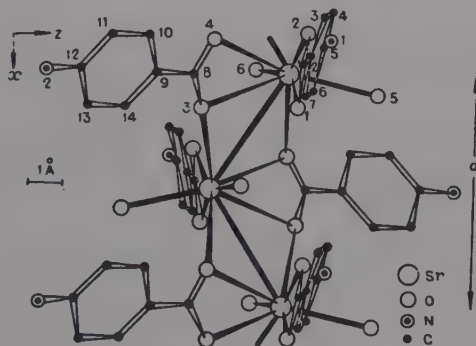


Fig. 1. The structure of  $\text{Sr}(\text{H}_2\text{NC}_6\text{H}_4\text{COO})_2\cdot 2\text{H}_2\text{O}$ .

## 2,4,6-TRINITROTOLUENE



W.R. CARPER, L.P. DAVIS and M.W. EXTINE, 1982. J. Phys. Chem., 86, 459-462.

Orthorhombic,  $\text{Pca}2_1$ ,  $a = 14.991$ ,  $b = 6.077$ ,  $c = 20.017$  Å,  $Z = 8$ . Cu radiation,  $R = 0.057$  for 1461 reflexions.

There are two independent molecules, A and B, in the asymmetric unit (Fig. 1). In the A form the 2, 4, and 6 nitro groups are twisted out of the benzene ring plane by  $51^\circ$ ,  $24^\circ$ , and  $43^\circ$ . The B-form nitro groups are twisted out of the plane by  $60^\circ$ ,  $30^\circ$ , and  $45^\circ$  in the 2, 4 and 6 positions.

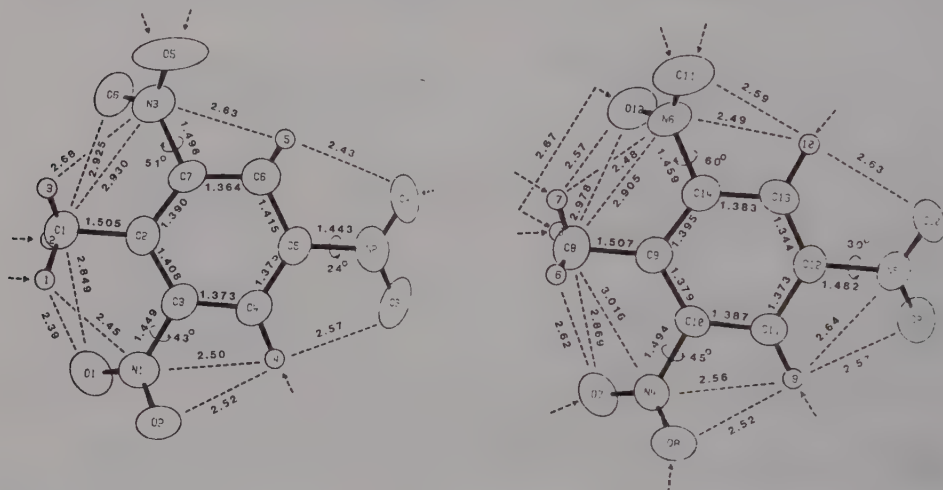
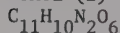


Fig. 1. The A and B molecules of 2,4,6-trinitrotoluene with intramolecular dimensions.

## ETHYL (Z)-2-NITRO-3-(4-NITROPHENYL)ACRYLATE



J.D. WALLIS and D.J. WATKIN, 1982. Acta Cryst., B38, 2057-2059.

Triclinic,  $\text{P}\bar{1}$ ,  $a = 7.803$ ,  $b = 7.860$ ,  $c = 10.961$  Å,  $\alpha = 77.19$ ,  $\beta = 82.16$ ,  $\gamma = 68.89^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.049$  for 1558 reflexions.

The two olefinic carbon atoms C(7) and C(8), (Fig. 1) and attached N and carbonyl group are coplanar; the olefinic nitro group and the benzene ring make angles of  $80.5(5)$  and  $9.3(5)^\circ$  with this plane. The tilt of the nitro group and a widening of the C(1)-C(7)-C(8) and C(2)-C(1)-C(7) angles to  $131.9(2)$  and  $1.251(2)^\circ$  respectively reduces the interaction between H(21) and O(4). The aromatic nitro group is twisted  $20.2(4)^\circ$  from the benzene ring plane. Molecules are stacked along the b-axis.

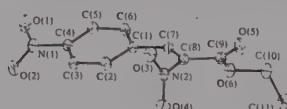


Fig. 1. The  $\text{C}_{11}\text{H}_{10}\text{N}_2\text{O}_6$  molecule.

## 2,4-DINITRODIPHENYL SULPHIDE



V. CODY and P.A. LEHMANN, 1982. *Cryst. Struct. Comm.*, **11**, 1671-1675.

Monoclinic,  $P2_1n$ ,  $a = 10.658$ ,  $b = 8.379$ ,  $c = 27.993$  Å,  $\beta = 100.49^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.081$  for 1751 reflexions.

The two independent molecules in the asymmetric unit have slightly different conformations (Fig. 1). The nitro groups are nearly coplanar with the phenyl ring and there are short intramolecular O...S contacts. Mean S-C bond lengths are 1.75(2) (to nitrophenyl) and 1.78 Å (to phenyl).

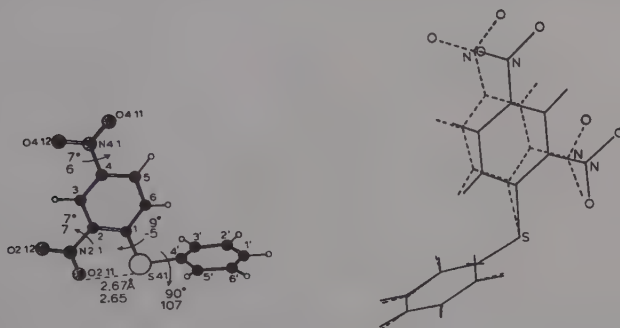


Fig. 1. The torsion angles in the two  $\text{C}_{12}\text{H}_8\text{N}_2\text{O}_4\text{S}$  molecules and a view of their (superimposed) conformations.

## 5-FLUORO-2,4,6-TRINITRO-1,3-BENZENEDIAMINE



H.L. AMMON, S.K. BHATTACHARJEE and J.R. HOLDEN, 1982. *Acta Cryst.*, **B38**, 1851-1854.

Monoclinic,  $Pc$ ,  $a = 7.5048$ ,  $b = 5.1195$ ,  $c = 11.7645$  Å,  $\beta = 99.625^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.04$  for 722 reflexions.

The nitro groups adjacent to F (Fig. 1) are rotated 17.8(8) and 23.2(8)° out of the ring plane giving a mean F...O distance of 2.45 Å; the third nitro group is rotated 3.4(8)°. All hydrogen atoms are involved in strong intramolecular hydrogen bonds to nitro oxygens and three also participate in weaker intermolecular hydrogen bonds which control crystal packing. The average C-C bond length for carbon bonded to amino groups is 1.434(9) and the average C-C-C bond angles at  $\text{NH}_2$ - and  $\text{NO}_2$ -substituted carbons are 116.5(6) and 121.4(6)° respectively. Some bond lengths are, C-F 1.329(7), C(2)-N(1) 1.462(9), C(4)-N(3) 1.430(9), C(6)-N(5) 1.456(8), C(3)-N(2) 1.315(8), C(5)-N(4) 1.320(9) Å.

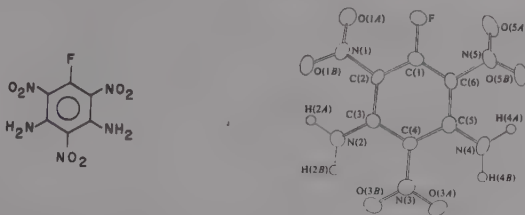


Fig. 1. The  $\text{C}_6\text{H}_4\text{FN}_5\text{O}_6$  molecule.

## 2,4-DINITROANILINE



L. PRASAD, E.J. GABE and Y. LE PAGE, 1982. *Acta Cryst.*, **B38**, 674-675.

Monoclinic,  $P2_1/c$ ,  $a = 8.171$ ,  $b = 12.863$ ,  $c = 7.513 \text{ \AA}$ ,  $\beta = 108.84^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.063$  for 785 reflexions.

Molecular dimensions are shown in Fig. 1. There is an intramolecular hydrogen bond,  $\text{N}(1)\text{-H}\cdots\text{O}(1)$  of length  $2.62 \text{ \AA}$  and molecules are linked by  $\text{N}(1)\text{-H}\cdots\text{O}(2)$  hydrogen bonds of length  $3.05 \text{ \AA}$ . The  $\text{N}(3), \text{O}(3), \text{O}(4)$  group is not involved in hydrogen bonding. The dihedral angles between the benzene ring plane and the amino,  $\text{N}(2)\text{O}(1)\text{O}(2)$  and  $\text{N}(3)\text{O}(3)\text{O}(4)$  groups are  $2.7$ ,  $4.3$  and  $6.6^\circ$  respectively.

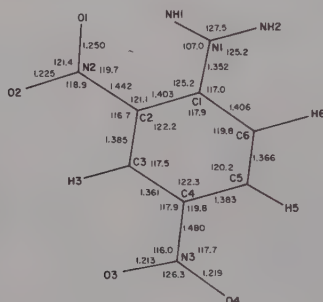


Fig. 1. Bond lengths and angles (e.s.d.'s  $0.005\text{-}0.006 \text{ \AA}$  and  $0.4^\circ$ ) in 2,4-dinitroaniline.

## p-NITROANILINE



M. COLAPIETRO, A. DOMENICANO, C. MARCIANTE and G. PORTALONE, 1982. *Z. Naturforsch.*, **37b**, 1309-1311.

Monoclinic,  $P2_1/n$ ,  $a = 12.337$ ,  $b = 6.037$ ,  $c = 8.597 \text{ \AA}$ ,  $\beta = 91.42^\circ$ ,  $D_m = 1.430$ ,  $Z = 4$ . Mo radiation,  $R = 0.044$  for 1171 reflexions.

This is a redetermination of the earlier reported structure (1). The molecular parameters are shown in Fig. 1. The dihedral angles between the benzene plane and the planes of the  $\text{NO}_2$  and  $\text{NH}_2$  groups are  $1.9(2)$  and  $7(2)^\circ$  respectively.

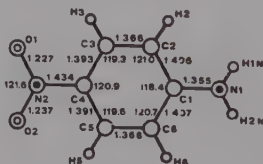
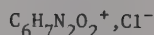
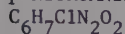


Fig. 1. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ) in p-nitroaniline. E.s.d.'s are  $0.002 \text{ \AA}$  and  $0.2^\circ$ .

1. *Structure Reports*, **26**, 696.

p-NITROANILINE HYDROCHLORIDE



G. PLOUG-SØRENSEN and E.K. ANDERSEN, 1982. *Acta Cryst.*, B38, 671-673.

Monoclinic,  $P2_1/c$ ,  $a = 4.9794$ ,  $b = 16.331$ ,  $c = 10.282$  Å,  $\beta = 115.01^\circ$ ,  $D_m = 1.519$ ,  $Z = 4$ . Mo radiation,  $R = 0.073$  for 1477 reflexions.

Layers of anilinium ions, perpendicular to the *a*-axis, are held together by ionic forces and hydrogen bonds to chloride ions (Fig. 1). The C(4),O(1),O(2),N(2) plane is twisted 7° from the ring plane and N(1) is 0.05 Å from the ring plane. Some bond lengths are, C(1)-N(1) 1.472(5), C(4)-N(2) 1.478(5), N(2)-O(1) 1.213(5), N(2)-O(2) 1.223(5), C-C (average) 1.378 Å.

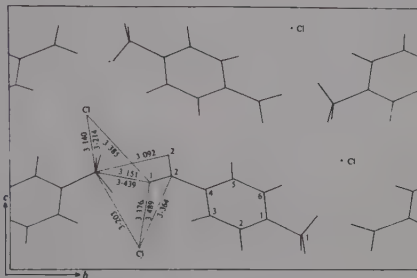


Fig. 1. The contents of the unit cell of p-nitroaniline hydrochloride. Hydrogen bonds and close contact distances are shown.

## ANILINE



M. FUKUYO, K. HIROTSU and T. HIGUCHI, 1982. *Acta Cryst.*, B38, 640-643.

Monoclinic,  $P2_1/c$ ,  $a = 21.822$ ,  $b = 5.867$ ,  $c = 8.386$  Å,  $\beta = 101.01^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.063$  for 834 reflexions (at 252 K).

Both N atoms in the two crystallographically independent molecules (Fig. 1) lie about 0.12 Å from the ring planes. The angle between the amino and ring planes averages 38(3)°. There are two N-H...N hydrogen bonds of length 3.180(6) and 3.373(5) Å involving only one H atom from each amino group. The sum of the bond angles around each N atom is 346 and 338°.

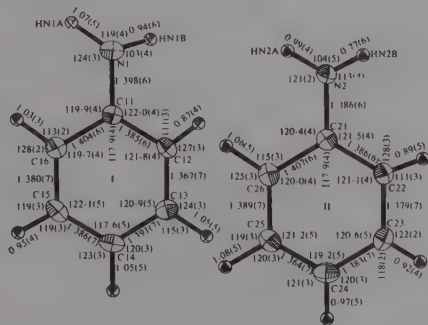
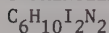


Fig. 1. Bond lengths and angles in aniline.



## o-PHENYLENEDIAMINE DIHYDROIODIDE



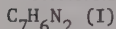
K. KOZAWA and T. UCHIDA, 1982. Bull. Chem. Soc. Jpn., 55, 943-944.

Orthorhombic, Pmmn,  $a = 7.828$ ,  $b = 13.158$ ,  $c = 5.043 \text{ \AA}$ ,  $D_m = 2.27$ ,  $Z = 2$ . Mo radiation,  $R = 0.052$  for 1359 reflexions.

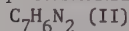
The compound is isostructural with the dihydrobromide salt (1). The cation is completely planar, with mm symmetry. Partial intercalation of iodide ions permits a relatively wide spacing between the cation molecular planes ( $5.043 \text{ \AA} = c\text{-axis}$ ).

1. Structure Reports, 38B, 135.

## m-CYANOANILINE



## p-CYANOANILINE



S. MERLINO and F. SARTORI, 1982. Acta Cryst., B38, 1476-1480.

I. Orthorhombic, Pna $2_1$ ,  $a = 8.27$ ,  $b = 16.94$ ,  $c = 4.89 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.065$  for 548 reflexions.

II. Monoclinic, P $2_1/c$ ,  $a = 7.35$ ,  $b = 5.56$ ,  $c = 16.10 \text{ \AA}$ ,  $\beta = 102.0^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.068$  for 768 reflexions.

In (I) N(1) is  $0.06$ , C(7)  $-0.077$  and N(2)  $-0.096 \text{ \AA}$  from the ring plane; corresponding distances in (II) are  $0.023$ ,  $0.073$  and  $0.175 \text{ \AA}$ . In (II) the  $\text{NH}_2$  group and the benzene ring are almost coplanar, but these groups are not coplanar in (I), (Fig.1). Molecules in both structures are linked by N-H...N hydrogen bonds; in (I) both H atoms of the amino group are involved while only one such H is involved in (II). Main bond lengths and angles are; in (I) C(2)-C(3)-C(4)  $121.2(6)^\circ$ , C(6)-C(1)-C(2)  $119.2(5)^\circ$ , C(3)-C(7)  $1.459(11)$ , C(1)-N(1)  $1.408(7) \text{ \AA}$ , in (II) C(3)-C(4)-C(5)  $119.2(3)^\circ$ , C-NH $_2$   $1.360(5)$ , C-CN  $1.430(5) \text{ \AA}$ . Bond lengths in (II) reveal a quinoid structure.

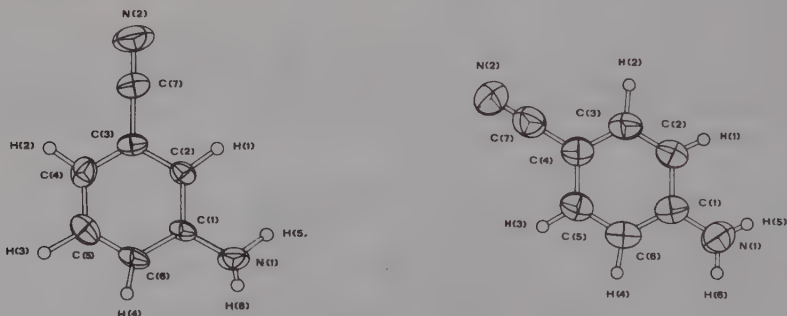


Fig. 1. The m-cyanoaniline (left) and p-cyanoaniline (right) molecules.

## p-TOLUIDINE HYDROCHLORIDE



M. COLAPIETRO, A. DOMENICANO and G. PORTALONE, 1982. Acta Cryst., B38, 2825-2829.

Monoclinic, P $2_1/c$ ,  $a = 9.116$ ,  $b = 9.357$ ,  $c = 9.913 \text{ \AA}$ ,  $\beta = 108.56^\circ$ ,  $D_m = 1.186$ ,  $Z = 4$ . Mo radiation,  $R = 0.0330$  for 1615 reflexions.

The crystal structure of p-toluidine hydrochloride has been determined previously (1). In the molecule the benzene ring has almost exact mm symmetry (Fig. 1). The  $\text{CH}_3, \text{C}_6\text{H}_4, \text{NH}_3^+$  and  $\text{Cl}^-$  ions are connected into infinite layers in the crystal through  $\text{Cl}^- \cdots \text{H-N}$  hydrogen bonds of length 3.12 to 3.15 Å.

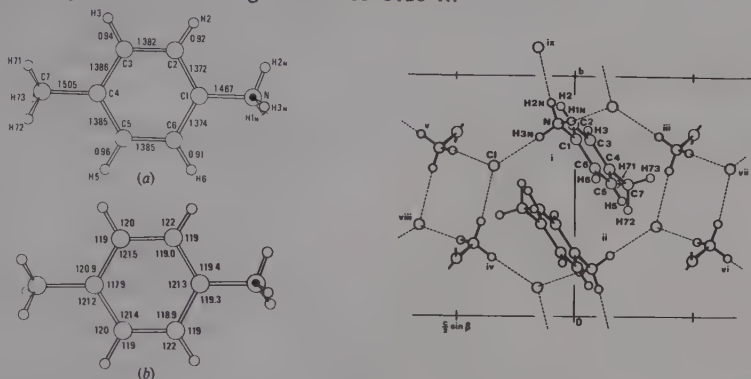


Fig. 1.  $\text{CH}_3, \text{C}_6\text{H}_4, \text{NH}_3^+ \text{Cl}^-$ : bond lengths and angles in the molecule and a projection of the crystal structure down a; broken lines represent intermolecular hydrogen bonds.

1. G. VON ELLER, 1955. Bull. Soc. Fr. Minéral. Cristallogr., 78, 275.

## 2-CHLORO-4-NITRO-N-(2-NITROVINYL)ANILINE $\text{C}_8\text{H}_6\text{ClN}_3\text{O}_4$

O. SIMONSEN, 1982. Acta Cryst., B38, 2060-2062.

Monoclinic,  $\text{P2}_1/\text{c}$ ,  $a = 8.7368$ ,  $b = 9.2068$ ,  $c = 13.655$  Å,  $\beta = 116.705^\circ$ ,  $D_m = 1.65$ ,  $Z = 4$ . Mo radiation,  $R = 0.055$  for 1389 reflexions.

The molecules are nearly planar with delocalisation of  $\pi$  electrons in the nitrovinyl moiety (Fig. 1). Planarity is stabilised by an intramolecular hydrogen bond ( $\text{N}(1) \cdots \text{O}(81)$  2.638(3) Å). The aromatic nitro group and the Schiff-base moiety make angles with the benzene ring of  $6.2(7)$  and  $7.6(9)^\circ$ . Molecules are stacked nearly parallel to the (04) planes with a short  $\text{Cl} \cdots \text{O}$  contact (3.082(3) Å).

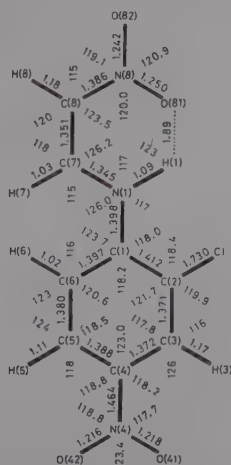
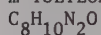


Fig. 1. Bond lengths and angles in the  $\text{C}_8\text{H}_6\text{ClN}_3\text{O}_4$  molecule.

## m-TOLYLUREA



M.R. CIAJOLO, F. LELJ, T. TANCREDI, P.A. TEMUSSI and A. TUZI, 1982. Gazz. Chim. Ital., 112, 429-430.

Orthorhombic, Pccn,  $a = 21.094$ ,  $b = 8.539$ ,  $c = 9.014$  Å,  $Z = 8$ . Cu radiation,  $R = 0.066$  for 538 reflexions.

The amide bond is trans (Fig. 1) and the torsion angle about N(1)-C(1) is  $57.4^\circ$ . Packing is determined by a network of hydrogen bonds.

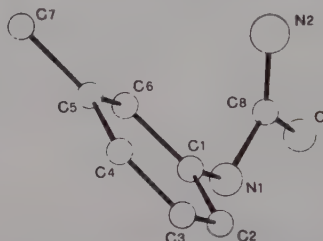
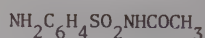
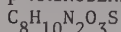


Fig. 1. The m-tolylurea molecule.

## p-AMINOBENZENE SULPHONACETAMIDE (SULPHACETAMIDE)



A.K. BASAK and S.K. MAZUMDAR, 1982. Cryst. Struct. Comm., 11, 1609-1616.

Tetragonal,  $P4_1$ ,  $a = 7.928$ ,  $c = 16.445$  Å,  $D_m = 1.385$ ,  $Z = 4$ . Mo radiation,  $R = 0.027$  for 958 reflexions.

The molecules (Fig. 1) are held in the crystal structure by a network of N-H...O hydrogen bonds ( $\text{N}\cdots\text{O}$  2.814-3.239(4) Å).

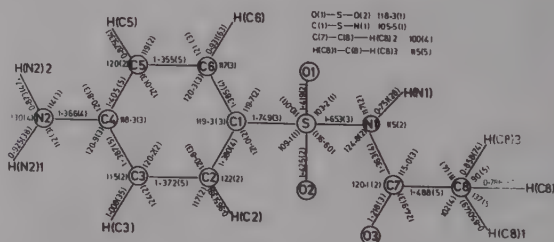
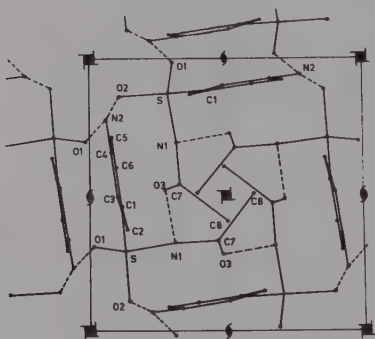
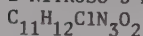


Fig. 1. The  $\text{C}_8\text{H}_{10}\text{N}_2\text{O}_3\text{S}$  molecule and packing.

## 2-NITROSO-3-METHYLAMINO-(p-CHLORO)CROTONALDEHYDE



A.C. VERONESE, G. CAVICCHIONI, P. SCRIMIN, G. VECCHIATI and V. BORTOLASI, 1982. Gazz. Chim. Ital., 112, 319-322.

Orthorhombic, Pbca,  $a = 18.376$ ,  $b = 11.762$ ,  $c = 10.982$  Å,  $Z = 8$ . Cu radiation,  $R =$

0.053 for 1588 reflexions.

The analysis shows that the molecule has a mesomeric polar form as the major contributor to the ground state structure (Fig. 1). The polar form is stabilized by charge transfer from the negatively charged oxygen atoms to the positively charged nitrogen atoms through the two short intramolecular hydrogen bonds. Some bond distances are N2-O2 1.290(3), N2-C8 1.344(3), C8-C7 1.483(4), C7-N1 1.343(3), C7-O1 1.236(3), C8-C9 1.445(4), C9-N3 1.301(3), HN1...O2 1.85(3), HN3...O1 1.92(4) Å.

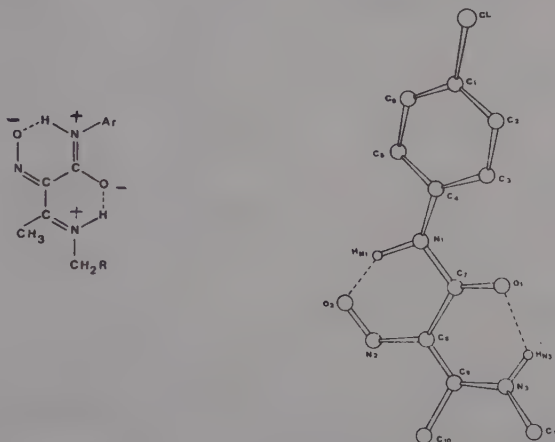
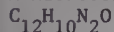


Fig. 1. The  $C_{11}H_{12}ClN_3O_2$  polar form and conformation.

#### N-NITROSODIPHENYLAMINE



A. BANERJEE, C.J. BROWN and J.F.P. LEWIS, 1982. *Acta Cryst.*, B38, 2744-2745.

Monoclinic,  $C2/c$ ,  $a = 16.283$ ,  $b = 8.827$ ,  $c = 16.508$  Å,  $\beta = 117.53^\circ$ ,  $D_m = 1.25$ ,  $Z = 8$ . Cu radiation,  $R = 0.067$  for 807 reflexions.

The structure comprises discrete molecules (Fig. 1) with no intermolecular interactions other than van der Waals forces. Bond distances are: mean C-C(benzene) 1.397, N-N 1.344(5) and N=O 1.206(7) Å.

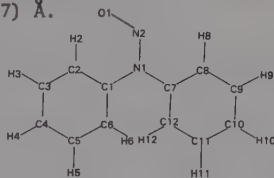
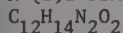


Fig. 1.  $C_{12}H_{10}N_2O$ : structural formula.

#### N-(2,2-DIACETYLVINYL)-o-PHENYLENEDIAMINE



C. SVENSSON, I. YMÉN and B. YOM-TOV, 1982. *Acta Chem. Scand.*, B36, 71-76.

Monoclinic,  $P2_1/c$ ,  $a = 10.259$ ,  $b = 10.988$ ,  $c = 10.896$  Å,  $\beta = 114.17^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.036$  for 1738 reflexions.

In the crystal the molecules are arranged in pairs around inversion centres. The approximately planar non-aromatic parts of the molecules overlap with an interplanar



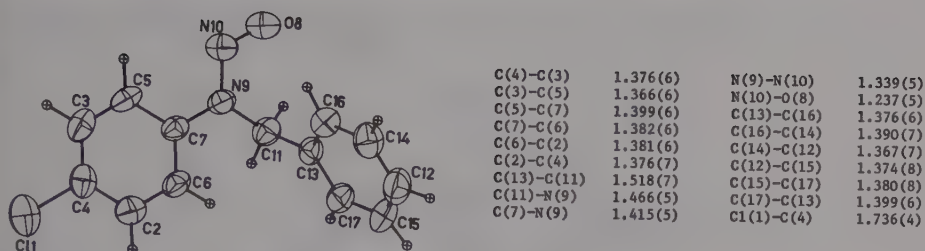


Fig. 1. The  $C_{13}H_{11}ClN_2O$  molecule and bond lengths.

N-(p-BROMOPHENYL)DIMETHACRYLAMIDE  
 $C_{14}H_{14}BrNO_2$

S.A. STONE-ELANDER, G.B. BUTLER, J.H. DAVIS and G.J. PALENIK, 1982. *Macromolecules*, **15**, 45-54.

Orthorhombic,  $Pbca$ ,  $a = 29.727$ ,  $b = 8.561$ ,  $c = 10.792$  Å,  $D_m = 1.492$ ,  $Z = 8$ . Cu radiation,  $R = 0.058$  for 1162 reflexions.

The compound was examined as part of a study of its polymerization by cobalt-60  $\gamma$ -rays. The imide group is in a trans-cis orientation (Fig. 1). Dihedral angle measurements indicated that O' was skewed out of the plane containing C7'-N-C7 by  $26.6^\circ$ , while O was skewed out by  $34.18^\circ$ . The phenyl ring is oriented in a plane with a dihedral angle of  $65^\circ$  to the imide plane, and, therefore, extended conjugation of the imide group through the phenyl ring would also be unlikely. An aspect of the monomer conformation and the most relevant to the polymerization process was the relative orientation of the two double bonds with respect to each other. The plane containing C9'-C8'-Me' and that containing C9-C8-Me were at an angle of  $87.45^\circ$  to each other. This nearly perpendicular arrangement would almost certainly allow no possibility for the through-space overlap of those  $\pi$  clouds. It was concluded that the monomer crystallized in a conformation that would favor the intramolecular over the intermolecular polymerization reaction.

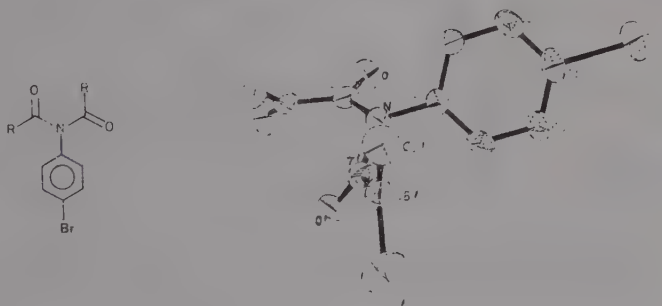


Fig. 1. The  $C_{14}H_{14}BrNO_2$  structure.

2-CHLORO-N-(2',6'-DIETHYLPHENYL)-N-(METHOXYMETHYL)ACETAMIDE (ALACHLOR)  
 $C_{14}H_{20}ClNO_2$

M.D. BUROW, B.A. KARCHER and R.A. JACOBSON, 1982. *Cryst. Struct. Comm.*, **11**, 747-750.

Monoclinic,  $P2_1/c$ ,  $a = 9.201$ ,  $b = 18.822$ ,  $c = 8.554$  Å,  $\beta = 100.71^\circ$ ,  $D_m = 1.13$ ,  $Z = 4$ . Mo radiation,  $R = 0.124$  for 1732 reflexions.



A view of this herbicide is shown in Fig. 1. There is large thermal motion of atoms at the periphery of the molecule. The N-C(13) distance (1.364(11) Å) is short.

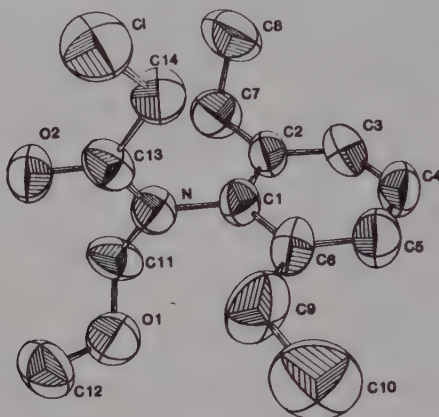
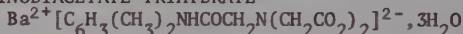
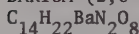


Fig. 1. A view of alachlor.

BARIUM (2,6-DIMETHYLPHENYL) CARBAMOYLMETHYLIMINODIACETATE TRIHYDRATE



R. FAGGIANI, C.J.L. LOCK, E. DEUTSCH, P. RICHARDS and S.C. SRIVASTAVA, 1982. Acta Cryst., B38, 733-736.

Monoclinic,  $P2_1/c$ ,  $a = 17.082$ ,  $b = 7.826$ ,  $c = 13.900$  Å,  $\beta = 99.26^\circ$ ,  $D_m = 1.73$ ,  $Z = 4$ . Mo radiation,  $R = 0.048$  for 3007 reflexions.

The structure (Fig. 1) comprises alternating layers of anions and water/ $\text{Ba}^{2+}$  cations in the (404) planes. Hydrogen bonding is important in bonding the structure. Bond distances and angles are normal.

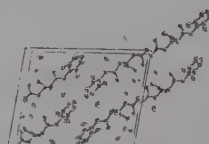
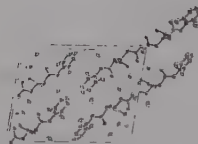
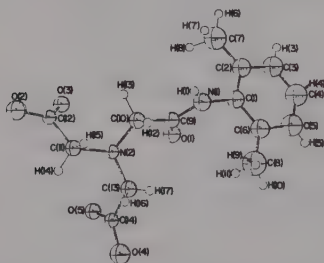
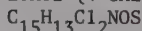


Fig. 1.  $\text{C}_{14}\text{H}_{22}\text{BaN}_2\text{O}_8$ : a view of the anion (left) and a stereoscopic view of packing within the unit cell (right).

ETHYL (4-CHLOROPHENYLIMINO)-4-CHLOROPHENYLMETHANE SULPHENATE



J.M.M. SMITS, J.H. NOORDIK, B.G. LENZ and B. ZWANENBURG, 1982. Cryst. Struct. Comm., 11, 1625-1628.

Monoclinic,  $C2/c$ ,  $a = 27.712$ ,  $b = 6.285$ ,  $c = 18.242$  Å,  $\beta = 94.70^\circ$ ,  $Z = 4$  [8]. Cu radiation,  $R = 0.040$  for 826 reflexions.

A view of this reaction product is shown in Fig. 1. Some bond lengths are S-O

1.632(5), S-C(1) 1.797(7), C(1)-N(1) 1.271(9), N(1)-C(5) 1.413(9) Å.

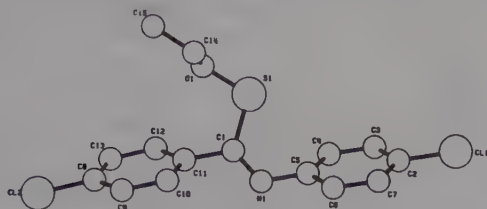
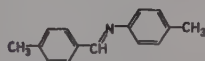


Fig. 1. The  $C_{15}H_{13}Cl_2NOS$  molecule.

p-METHYL-N-(p-METHYLBENZYLIDENE)ANILINE (FORM I)  
 $C_{15}H_{15}N$



I. BAR and J. BERNSTEIN, 1982. *Acta Cryst.*, **B38**, 121-125.

Monoclinic,  $P2_1/c$ ,  $a = 6.089$ ,  $b = 7.751$ ,  $c = 26.766$  Å,  $\beta = 103.16^\circ$ ,  $D_m = 1.12$ ,  $Z = 4$ .  
 Cu radiation,  $R = 0.075$  for 1374 reflexions.

Bond lengths and angles in this structure (Form I) of the title compound (Fig. 1) agree well with those of the other two polymorphs, Forms II (1) and III (2). The molecular conformation differs significantly from the conformation of Form II, but agrees with the planar conformation of Form III. The mode of disorder observed here differs from those of previously determined benzylideneanilines.

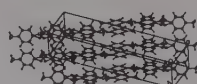
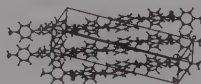
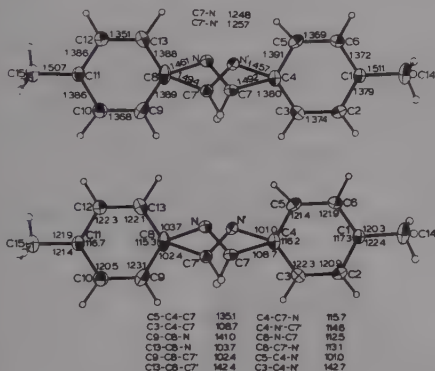


Fig. 1.  $C_{15}H_{15}N$ : bond lengths and angles; bridge atoms N, C(7) and the hydrogen attached to C(7) are disordered as shown.

1. *Structure Reports*, **43B**, 137.

2. *Ibid*, **42B**, 84.

p-TRICYANOVINYLN-N-ETHYL-N-(β-CYANOETHYL)ANILINE  
 $C_{16}H_{13}N_5$

Z.P. POVET'EVA, L.A. CHETKINA and B.P. BESPALOV, 1982. *Zh. Strukt. Khim.*, **23**-2, 168-171 [*J. Struct. Chem.*, **23**, 321-324].

Monoclinic,  $P2_1/b$ ,  $a = 6.180$ ,  $b = 18.347$ ,  $c = 12.956$  Å,  $\gamma = 93.26^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.051$  for 1153 reflexions.

Bond lengths and angles ( $\sigma = 0.006-0.007$  Å,  $0.4-0.6^\circ$  for non-hydrogen atoms) are shown in Fig. 1. The six-membered ring is planar to within 0.003 Å and the tricyano-

vinyl group to within 0.015 Å, the angle between the two mean planes being 3.3°. Angular deformations at C(4) and C(7) may be due to the steric interaction C(11)...H(3) 2.37 Å.

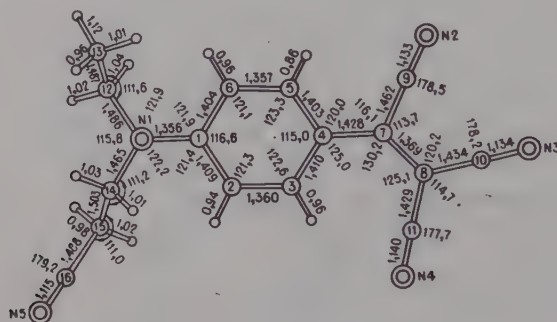


Fig. 1. The  $C_{16}H_{13}N_5$  molecule.

1,6-BIS(p-CHLOROPHENYL)-3,4-DIACETYL-HEXA-AZA-1,5-DIENE  
 $C_{16}H_{14}Cl_2N_6O_2$

D. MACKAY, D.D. McINTYRE and N.J. TAYLOR, 1982. *J. Org. Chem.*, **47**, 532-535.

Monoclinic,  $I2/c$ ,  $a = 13.863$ ,  $b = 8.985$ ,  $c = 14.763$  Å,  $\beta = 94.37^\circ$ ,  $D_m = 1.42$ ,  $Z = 4$ . Mo radiation,  $R = 0.032$  for 1682 reflexions.

The molecule has  $C_2$  symmetry in the crystal, and consists of two planar halves connected through the central N(1)-N(1)' bond with a dihedral angle of  $84.4^\circ$  (Fig. 1). The C(3)-N(3) bond is considerably displaced from the ring bisector, a steric effect allowing approximately equal N(3)...H(4) and N(2)...H(8) interactions. The C(1)-N(1) acetyl-amide bond is notably long, 1.403(2) Å, and the carbonyl bond is correspondingly short at 1.200(2) Å. These dimensions and an unusually high carbonyl stretching frequency indicate strong electrophilic character in the acetyl group of this molecule.

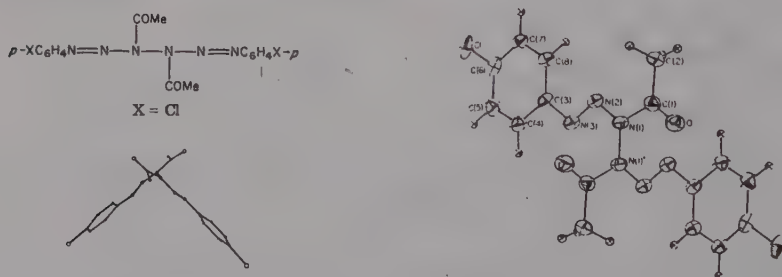


Fig. 1. Formula and views of the hexa-aza-diene molecule,  $C_{16}H_{14}Cl_2N_6O_2$ .

2-[(2-METHYLACETAMIDO-5-CHLOROPHENYL)-2-NITROPHENYLMETHYLENIMINO]ETHANOL  
 $C_{18}H_{18}ClN_3O_4$

K. HARANO, M. YASUDA, Y. ARIYOSHI, M. MAEDA and M. KOJIMA, 1982. *Cryst. Struct. Comm.*, **11**, 1525-1530.

Monoclinic,  $P2_1/a$ ,  $a = 14.508$ ,  $b = 14.830$ ,  $c = 8.289$  Å,  $\beta = 94.42^\circ$ ,  $D_m = 1.384$ ,  $Z = 4$ . Mo radiation,  $R = 0.069$  for 2362 reflexions.

The analysis establishes the structure of this reduction product to be as

shown in Fig. 1 with the 2-hydroxyethyl and 2-nitrophenyl groups cis. The C15=O16 (1.311 Å) and N13-C15 (1.266 Å) are longer and shorter respectively than anticipated consistent with significant  $\pi$ -orbital overlap in the N-C=O system.

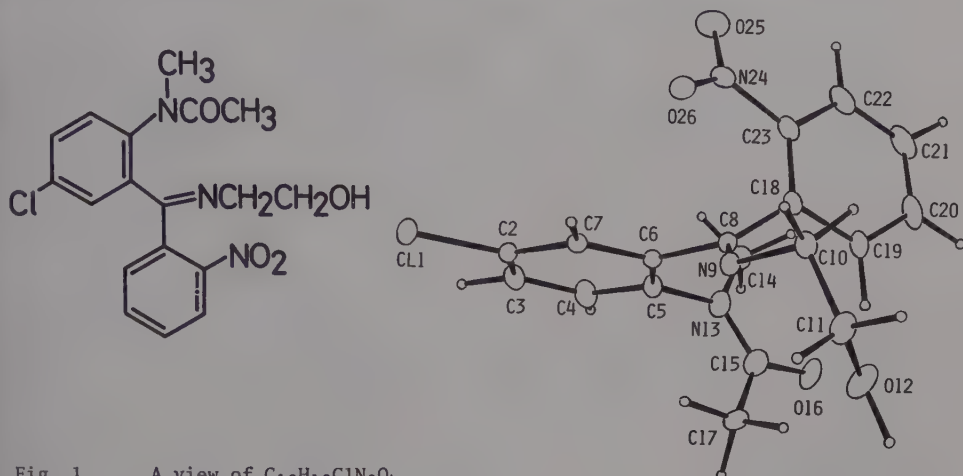
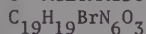


Fig. 1. A view of  $C_{18}H_{18}ClN_3O_4$ .

6'-ACETAMIDO-6-BROMO-2-CYANO-4'-DIETHYLAMINO-4-NITROAZOBENZENE



J.G. HANDAL, R.P. GRUSKA, M. SHOJA and J.G. WHITE, 1982. Z. Krist., **161**, 61-67.

Monoclinic,  $P2_1/c$ ,  $a = 7.070$ ,  $b = 12.409$ ,  $c = 23.439$  Å,  $\beta = 101.35^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.078$  for 2743 reflexions.

The conformation in the crystal is one with the acetamide group trans to the Br atom (Fig. 1) and is apparently stabilized by intramolecular bifurcated N-H...N hydrogen bonding.

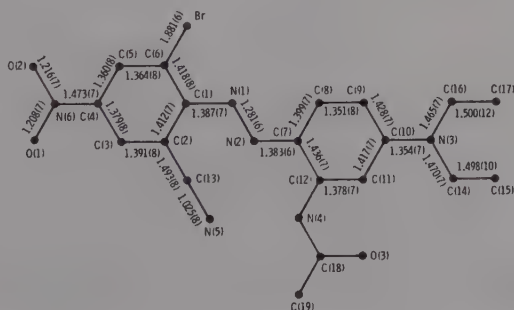
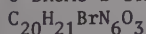


Fig. 1. The  $C_{19}H_{19}BrN_6O_3$  molecule and bond lengths.

6-BROMO-2-CYANO-4'-DIETHYLAMINO-4-NITRO-6'-PROPIONAMIDOAZOBENZENE



J.G. HANDAL, M. SHOJA and J.G. WHITE, 1982. Z. Krist., **161**, 231-236.

Monoclinic,  $C2/c$ ,  $a = 29.015$ ,  $b = 10.867$ ,  $c = 24.945$  Å,  $\beta = 147.80^\circ$ ,  $D_m = 1.50$ ,  $Z = 8$ . Cu radiation,  $R = 0.091$  for 2364 reflexions.

The conformation in the crystal has the propionamide group trans to the Br atom (Fig. 1) and is apparently stabilized by bifurcated  $\text{NH}\dots\text{N}$  hydrogen bonding.

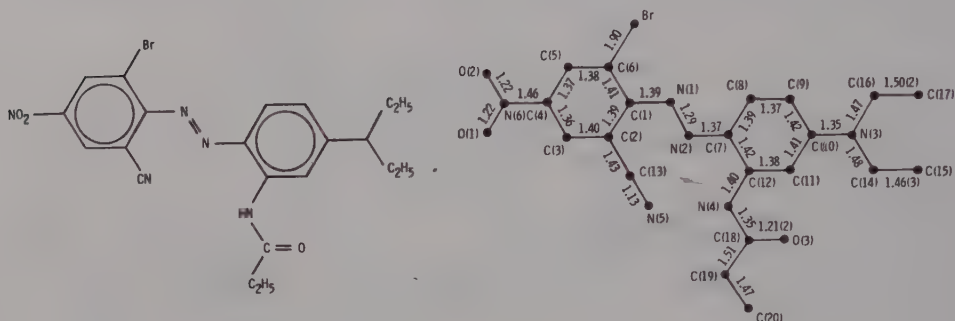


Fig. 1. The  $\text{C}_{20}\text{H}_{21}\text{BrN}_6\text{O}_3$  molecule and bond lengths ( $\sigma$  0.01 Å).

### TRI-p-TOLYLAMINE

$\text{C}_{21}\text{H}_{21}\text{N}$

S.L. REYNOLDS and R.P. SCARINGE, 1982. Cryst. Struct. Comm., **11**, 1129-1134.

Triclinic,  $\text{P}\bar{1}$ ,  $a = 12.403$ ,  $b = 10.872$ ,  $c = 12.944$  Å,  $\alpha = 88.48$ ,  $\beta = 103.34$ ,  $\gamma = 88.93^\circ$ ,  $D_m = 1.10$ ,  $Z = 4$ . Cu radiation,  $R = 0.043$  for 5732 reflexions.

The two independent molecules have similar conformations. The N atom (Fig. 1) has near trigonal-planar geometry.

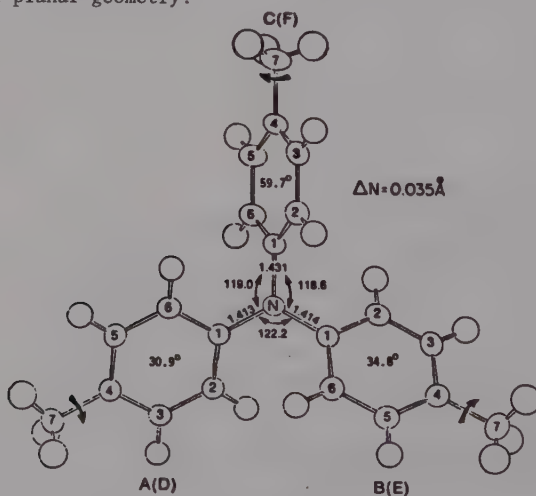


Fig. 1. A  $\text{C}_{21}\text{H}_{21}\text{N}$  molecule with mean dimensions and twist angles.

(aR,1'R,1''R) -N-(1-METHYL-6-PHENYL-4-OXO-3-OXA-4-AZAHEPT-1-YL) -N-CHLOROACETYL-2-ETHYL-6-METHYLANILINE

$\text{C}_{23}\text{H}_{29}\text{ClN}_2\text{O}_3$  (I)

(a*S*,1'*R*,1''*R*)-N-(1-METHYL-6-PHENYL-4-OXO-3-OXA-4-AZAHEPT-1-YL)-N-CHLOROACETYL-2-ETHYL-6-METHYLANILINE  
 $C_{23}H_{29}ClN_2O_3$  (II)

H. MOSER, G. RIHS and H. SAUTER, 1982. *Z. Naturforsch.*, **37b**, 451-462.

I. Monoclinic,  $P2_1$ ,  $a = 10.915$ ,  $b = 9.252$ ,  $c = 13.139$  Å,  $\beta = 121.02^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.072$  for 2152 reflexions.

II. Orthorhombic,  $P2_12_12_1$ ,  $a = 9.568$ ,  $b = 15.111$ ,  $c = 15.490$  Å,  $Z = 4$ . Cu radiation,  $R = 0.113$  for 1851 reflexions.

The analysis established the structures shown in Fig. 1 with normal values for their bond lengths and angles.

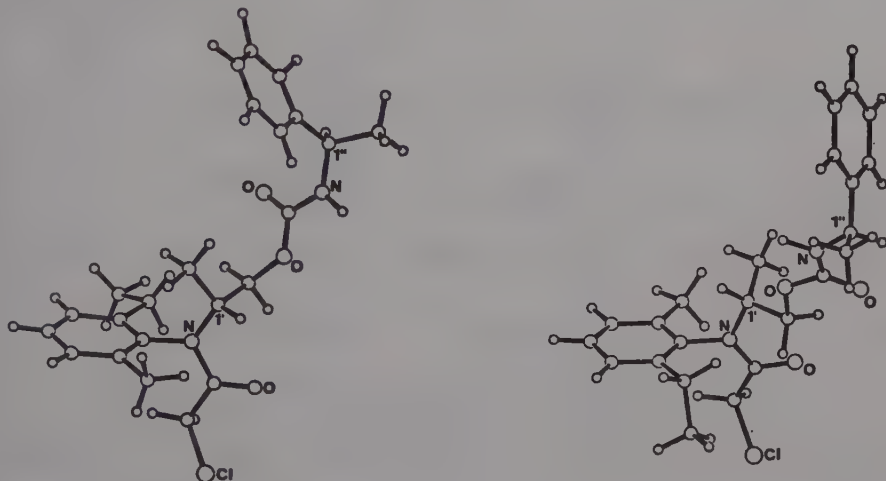


Fig. 1. The structures of the (a*R*,1'*R*,1''*R*) (left) and (a*S*,1'*R*,1''*R*) (right) isomers of  $C_{23}H_{29}ClN_2O_3$ .

#### BIS(4-BIPHENYLYL)SULPHUR DIIMIDE

$C_{24}H_{18}N_2S$

V. BUSETTI, 1982. *Acta Cryst.*, **B38**, 665-667.

Monoclinic,  $Cc$ ,  $a = 6.116$ ,  $b = 10.630$ ,  $c = 28.723$  Å,  $\beta = 91.30^\circ$ ,  $D_m = 1.29$ ,  $Z = 4$ . Mo radiation,  $R = 0.063$  for 1108 reflexions.

In the molecule (Fig. 1) the phenyl ring planes A (C(1) to C(6)), B (C(7) to C(12)), C (C(13) to C(18)) and D (C(19) to C(24)) are twisted such that  $A \wedge B$  is  $37.1^\circ$  and  $C \wedge D$  is  $39.8^\circ$ . C(1) and C(13) are 0.05 and 0.03 Å from the  $N_2S$  plane. The torsion angles C(13)-N(2)-S(1)-N(1) and C(1)-N(1)-S(1)-N(2) are  $-178$  and  $-4^\circ$ .





## Phase II:

Monoclinic,  $P2_1/c$ ,  $a = 5.181$ ,  $b = 36.629$ ,  $c = 6.827$  Å,  $\beta = 113.17^\circ$ ,  $D_m = 1.203$ ,  $Z = 2$ . Cu radiation,  $R = 0.038$  for 1616 reflexions.

Two solid-state polymerizable monomer phases are obtained for the unresolved diacetylene having the chiral substituent group  $-\text{CH}_2\text{HC}^*(\text{CH}_2\text{CH}_3)\text{OCONHC}_6\text{H}_5$ . For phase I (Fig. 1), chiral molecules react by 1,4-addition polymerization of glide-related neighbors having opposite handedness to provide a regular backbone structure in which nearest-neighbor substituent groups have different chirality, but next-nearest-neighbor groups have the same chirality. Centrosymmetric diacetylene molecules present in the racemic phase II (Fig. 2) react by 1,4-polymerization with translation-related neighbors. Phase I polymerizes by solid solution formation to provide a polymer crystal, while phase II polymer is ordered only in chain-axis projection. Bond lengths ( $\sigma = 0.004$ – $0.03$  Å for I,  $0.002$ – $0.004$  Å for II) and angles are as expected.

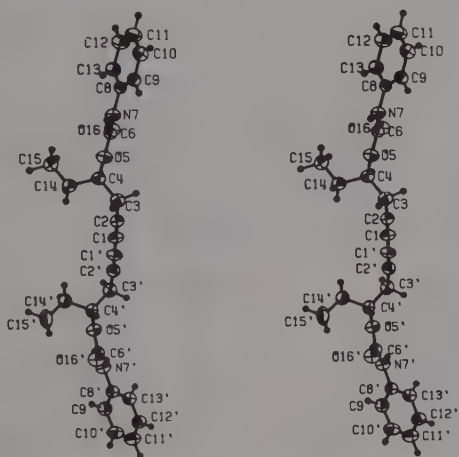


Fig. 1. Stereoview of the phase I molecule of  $\text{C}_{26}\text{H}_{28}\text{N}_2\text{O}_4$ .

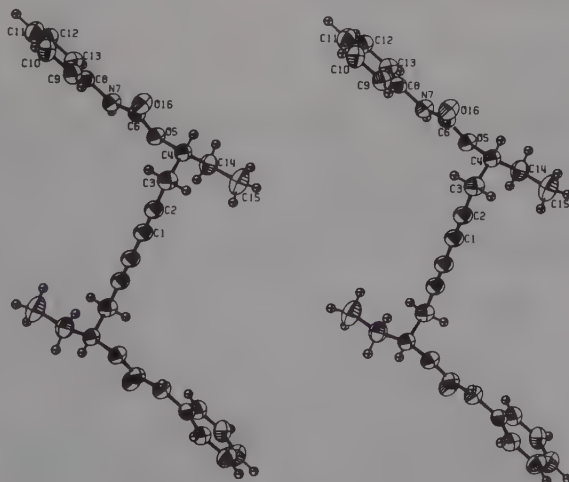
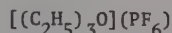


Fig. 2. Stereoview of the phase II molecule of  $\text{C}_{26}\text{H}_{28}\text{N}_2\text{O}_4$ .

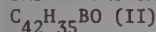


In these isomorphous structures (Fig. 1) the benzene ring lies on a twofold axis and shows angular distortion. One nitro group in each structure has a large rotation angle ( $67(0.8)$  in I and  $63(0.8)^\circ$  in II). The water molecules are located on the triad axes and are weakly hydrogen-bonded in 'tunnels'.

## TRIETHYLOXONIUM HEXAFLUOROPHOSPHATE



## TRIPHENYLOXONIUM TETRAPHENYLBORATE



M.I. WATKINS, W.M. IP, G.A. OLAH and R. BAU, 1982. J. Am. Chem. Soc., 104, 2365-2372.

I. Orthorhombic, Pnma,  $a = 16.475$ ,  $b = 9.965$ ,  $c = 6.557 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.058$  for 618 reflexions (at  $-50^\circ\text{C}$ ).

II. Monoclinic,  $P2_1/n$ ,  $a = 21.101$ ,  $b = 11.107$ ,  $c = 13.603 \text{ \AA}$ ,  $\beta = 90.75^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.048$  for 1379 reflexions.

The structures of the two oxonium cations are shown in Fig. 1. The  $\text{Et}_3\text{O}^+$  cation is established as being pyramidal, while the  $\text{Ph}_3\text{O}^+$  cation was found to be nearly planar. The average O-C distances are  $1.499(10)$  in I and  $1.472(9) \text{ \AA}$  in II. The phenyl rings in II are tilted by an average of  $59.7^\circ$  from the  $\text{OC}_3$  plane, indicating a minimal p- $\pi$  interaction between O and its aromatic ligands. Anion geometry is normal.

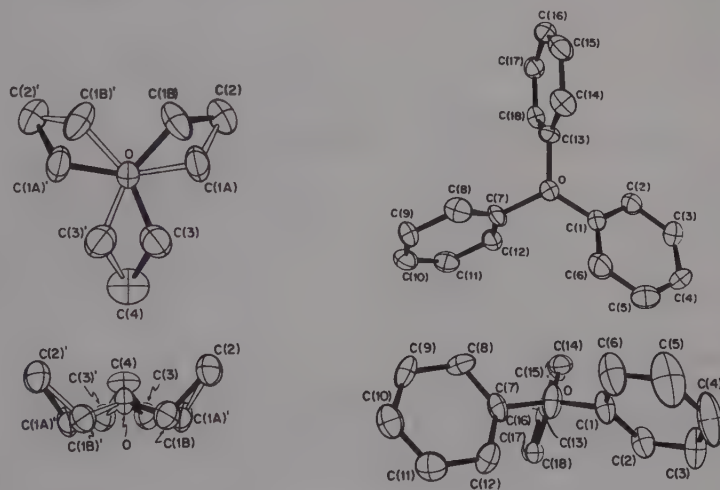
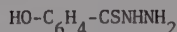


Fig. 1. Top and side views of the disordered  $\text{Et}_3\text{O}^+$  cation (left) and the  $\text{Ph}_3\text{O}^+$  cation (right).

## o-HYDROXYBENZOTHIODIAZIDE

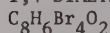


S. PRASAD, 1982. Z. Krist., 159, 169-172.

Monoclinic,  $P2_1/a$ ,  $a = 14.978$ ,  $b = 5.911$ ,  $c = 9.288 \text{ \AA}$ ,  $\beta = 109.5^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.17$  for 375 reflexions.

The analysis is of low accuracy but dimensions indicate that the compound is the thiol rather than thione form. The C-S distance is  $1.77(3)$  and N-N  $1.40(4) \text{ \AA}$ .

## 1,4-DIMETHOXY-2,3,5,6-TETRABROMOBENZENE



M.W. WIECZOREK, 1982. Cryst. Struct. Comm., 11, 1169-1173.

Monoclinic,  $P2_1/n$ ,  $a = 4.228$ ,  $b = 14.949$ ,  $c = 9.260$  Å,  $\beta = 100.76^\circ$ ,  $D_m = 2.61$ ,  $Z = 2$ . Mo radiation,  $R = 0.056$  for 847 reflexions.

The molecule (Fig. 1) lies on a crystallographic inversion centre. Deviations of atoms from the benzene ring plane are Br(1) 0.010(1), Br(2) 0.006(1), O -0.076(7) Å. The dihedral angle between the benzene ring plane and that through C(3), O, and C(1) is  $87.2(1)^\circ$ .

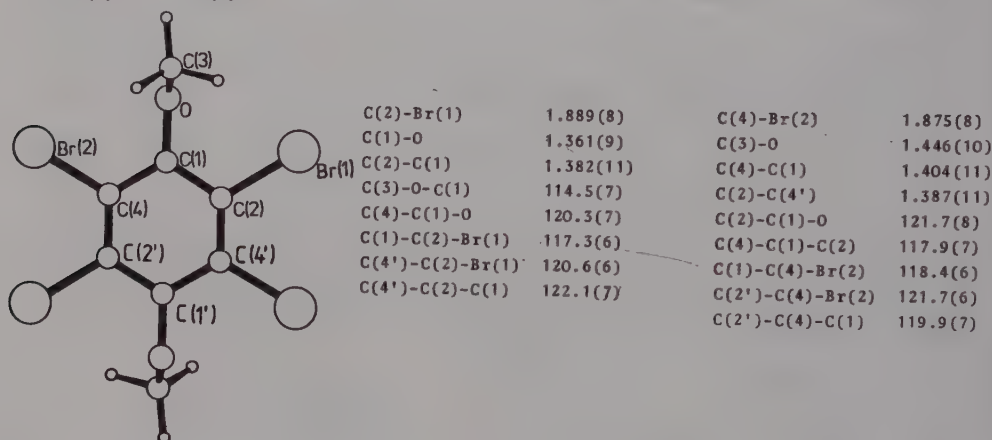
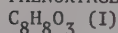
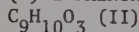
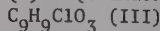
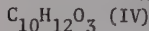


Fig. 1. Perspective view of  $\text{C}_8\text{H}_6\text{Br}_4\text{O}_2$  and dimensions.

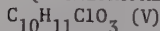
## PHENOXYACETIC ACID

( $\pm$ )-2-PHENOXYPROPIONIC ACID( $\pm$ )-2-(4-CHLOROPHENOXY)PROPIONIC ACID

## 2-METHYL-2-PHENOXYPROPIONIC ACID



## 2-(4-CHLOROPHENOXY)-2-METHYLPROPIONIC ACID



C.H.L. KENNARD, G. SMITH and A.H. WHITE, 1982. Acta Cryst., B38, 868-875.

I. Monoclinic,  $P2_1/c$ ,  $a = 12.39$ ,  $b = 5.114$ ,  $c = 11.71$  Å,  $\beta = 91.36^\circ$ ,  $D_m = 1.36$ ,  $Z = 4$ . Mo radiation,  $R = 0.046$  for 869 reflexions.

II. Monoclinic,  $A2/a$ ,  $a = 11.07$ ,  $b = 5.275$ ,  $c = 29.22$  Å,  $\beta = 98.17^\circ$ ,  $D_m = 1.29$ ,  $Z = 8$ . Mo radiation,  $R = 0.059$  for 934 reflexions.

III. Monoclinic,  $A2/a$ ,  $a = 11.533$ ,  $b = 5.167$ ,  $c = 31.91$  Å,  $\beta = 99.35^\circ$ ,  $D_m = 1.41$ ,  $Z = 8$ . Mo radiation,  $R = 0.051$  for 1165 reflexions.

IV. Monoclinic,  $P2_1/n$ ,  $a = 14.099$ ,  $b = 5.698$ ,  $c = 11.700$  Å,  $\beta = 91.19^\circ$ ,  $D_m = 1.26$ ,  $Z = 4$ . Mo radiation,  $R = 0.055$  for 1287 reflexions.

V. Monoclinic,  $P2_1/n$ ,  $a = 21.127$ ,  $b = 7.966$ ,  $c = 6.329$  Å,  $\beta = 90.05^\circ$ ,  $D_m = 1.32$ ,  $Z = 4$ . Mo radiation,  $R = 0.060$  for 955 reflexions.

All the acids (Fig. 1) form hydrogen-bonded cyclic dimers with O-H...O distances of 2.629 (I), 2.664 (II), 2.657 (III), 2.665 (IV) and 2.627 (V) Å. Phenoxyacetic acid (I) is essentially a planar molecule while in the propionic acids (II) and (III) (which are structurally similar but not isomorphous to each other), the  $\beta$ -methyl group lies approximately in the plane of the phenoxy group, with the plane of the carboxylic acid residue synclinally related in a manner similar to all known 2-phenoxypropionic acids. Acids (IV) and (V) are conformationally similar to (II) and (III) in this respect but possess a different overall conformational motif for the oxo-alkanoic acid side chain due to the steric effects of the additional methyl group on the  $\alpha$  carbon. Phenoxyacetic acid exhibits disorder in the carboxylic acid group with the two C-O distances equal and the proton disordered in the O...H...O hydrogen bond. No such disorder was found for the other acids.

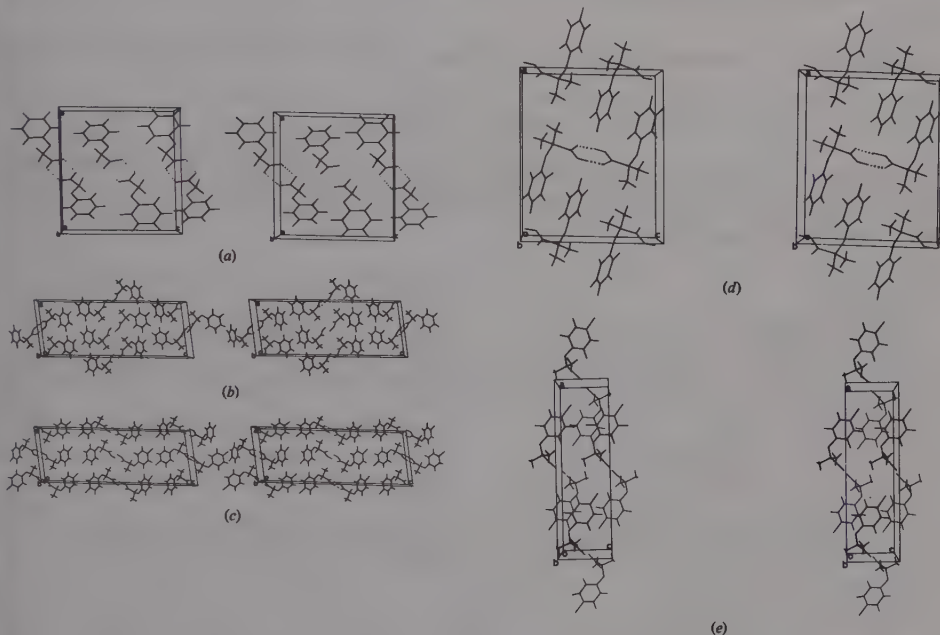


Fig. 1. Stereoscopic views of the packing of the molecules in (a) phenoxyacetic acid (I), (b)  $(\pm)$ -2-phenoxypropionic acid (II), (c)  $(\pm)$ -2-(4-chlorophenoxy)propionic acid (III), (d) 2-methyl-2-phenoxypropionic acid (IV) and (e) 2-(4-chlorophenoxy)-2-methylpropionic acid (V).

#### 2-HYDROXY-5-METHYL-ISOPHTHALALDEHYDE



T. RAY and S.P. SEN GUPTA, 1982. Cryst. Struct. Comm., 11, 59-63.

Orthorhombic,  $P2_12_12_1$ ,  $a = 25.998$ ,  $b = 7.555$ ,  $c = 3.944$  Å,  $Z = 4$ . Mo radiation,  $R = 0.092$  for 476 reflexions.

The molecule (Fig. 1) has an intramolecular hydrogen bond (O...O 2.62 Å).



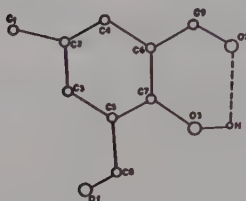


Fig. 1. A view of  $C_9H_9O_3$ .

## 2-(4-CHLOROPHENOXY)PROPIONIC ACID



S. RAGHUNATHAN, K. CHANDRASEKHAR and V. PATTABHI, 1982. *Acta Cryst.*, B38, 2536-2538.

Monoclinic,  $C2/c$ ,  $a = 31.858$ ,  $b = 5.171$ ,  $c = 11.535$  Å,  $\beta = 99.312^\circ$ ,  $D_m = 1.42$ ,  $Z = 8$ .  
Co radiation,  $R = 0.074$  for 1002 reflexions. [See also (1).]

Bond lengths and angles in the molecule (Fig. 1) are generally as expected. The angle between the benzene ring and the carboxyl-group plane is  $81.9(8)^\circ$ . The molecules are linked in the crystal into dimers by centrosymmetric  $O-H \cdots O$  hydrogen bonds, with  $O \cdots O$  2.683(9) Å.

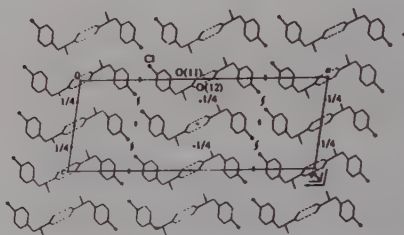
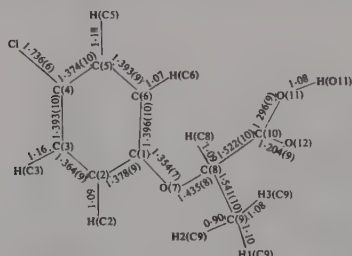


Fig. 1.  $C_9H_9ClO_3$ : bond lengths in the molecule and a view of the structure projected down  $b$ ; hydrogen bonds are shown by broken lines.

1. This volume, p96.

## p-METHYLPHENOXYACETIC ACID



S.V. KUMAR and L.M. RAO, 1982. *Z. Krist.*, 161, 45-51.

Monoclinic,  $P2_1/c$ ,  $a = 13.868$ ,  $b = 5.153$ ,  $c = 11.896$  Å,  $\beta = 102.17^\circ$ ,  $D_m = 1.326$ ,  $Z = 4$ . Cu radiation,  $R = 0.048$  for 784 reflexions.

A refinement with new data of a structure reported previously (1). The molecules pack as centrosymmetric dimers  $O \cdots O$  2.610(5) Å; the bond lengths (Fig. 1) and the presence of two hydrogen peaks at the carboxyl hydrogen bond are indicative of disorder of the carboxyl oxygen atoms.

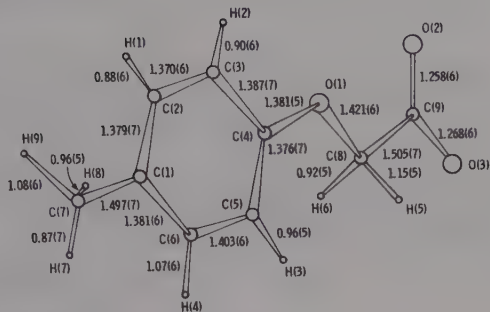


Fig. 1. Bond lengths in  $C_9H_{10}O_3$ .

1. Structure Reports, 48B, 114.

2-METHYL-2-(2,4,5-TRICHLOROPHENOXY)PROPIONIC ACID



C.H.L. KENNARD, G. SMITH, W.L. DUAX and D.C. SWENSON, 1982. Aust. J. Chem., 35, 2145-2149.

Triclinic,  $P\bar{1}$ ,  $a = 9.859$ ,  $b = 11.542$ ,  $c = 6.247$  Å,  $\alpha = 100.13$ ,  $\beta = 97.26$ ,  $\gamma = 114.68^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.051$  for 1249 reflexions.

The molecules (Fig. 1) form centrosymmetric hydrogen-bonded dimers ( $O(11)-H\cdots O(10')$  2.638(5) Å) and adopt the synplanar-synplanar conformation. The bond lengths have normal values ( $O(7)-C(1), C(8)$  1.380(6), 1.450(6) Å).

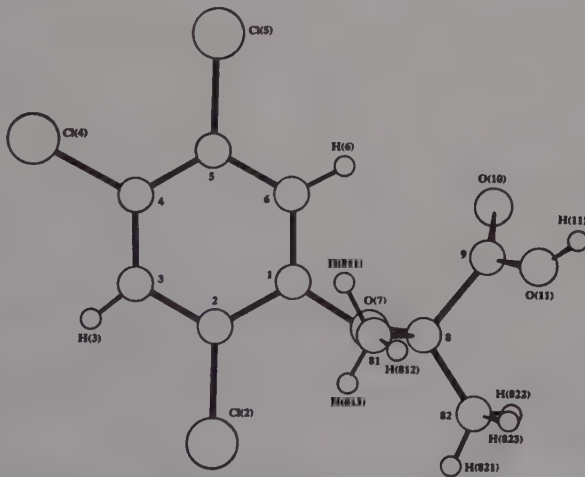


Fig. 1. Molecular conformation of 2-methyl-2-(2,4,5-trichlorophenoxy)propionic acid.

2-ETHOXY-cis-CINNAMIC ACID



R.F. BRYAN and P. HARTLEY, 1982. J. Chem. Soc. Perkin II, 191-193.

Orthorhombic,  $Pbca$ ,  $a = 10.811$ ,  $b = 12.137$ ,  $c = 15.223$  Å,  $D_m = 1.27$ ,  $Z = 8$ . Cu radiation,  $R = 0.041$  for 1380 reflexions.

There is strain about the *cis* double bond (Fig. 1) resulting in expanded bond angles,  $C(1)-C(9)-C(8)$   $129.8^\circ$ ,  $C(9)-C(8)-C(7)$   $126.3^\circ$  and  $C(8)-C(7)-O(1)$   $124.2^\circ$ . The plane of the double bond is inclined to the phenyl ring at  $59.4^\circ$  and to the carboxyl group at  $12.3^\circ$ . The degree of twist about  $C(8)-C(7)$  and  $C(9)-C(1)$  is limited by the intramolecular contact between  $O(1)$  and  $C(6)$  of  $3.10$  Å. The acid is present in the crystal as centrosymmetric hydrogen bonded dimers with  $O-H \cdots O$   $2.651$  Å. In no case do both atoms of a given double bond come closer than  $6$  Å to both of the corresponding atoms of a neighbouring molecule in the crystal.

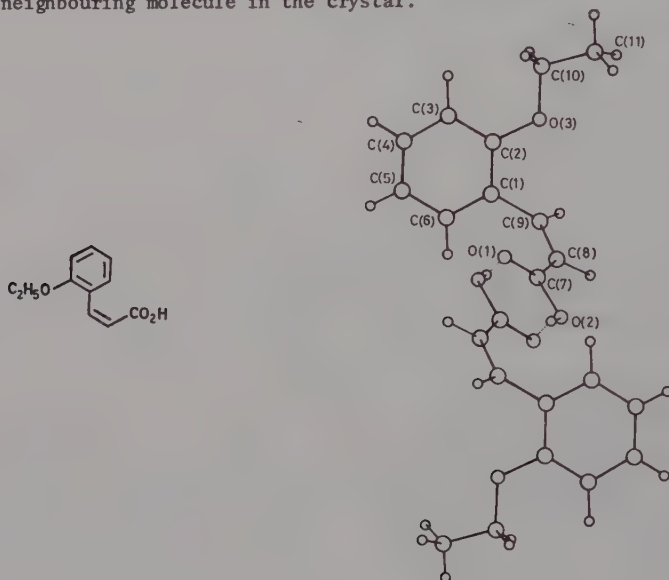


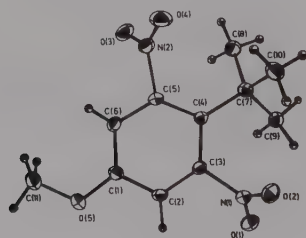
Fig. 1. Molecular skeleton and structure of  $C_{11}H_{12}O_3$ .

#### 4-t-BUTYL-3,5-DINITROANISOLE $C_{11}H_{14}N_2O_5$

W. VAN HAVERE, A.T.H. LENSTRA and H.J. GEISE, 1982. *Acta Cryst.*, **B38**, 3119-3120.

Triclinic,  $P\bar{1}$ ,  $a = 8.887$ ,  $b = 10.148$ ,  $c = 8.024$  Å,  $\alpha = 110.49^\circ$ ,  $\beta = 112.15^\circ$ ,  $\gamma = 89.58^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.029$  for 1617 reflexions.

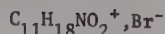
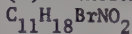
Severe crowding (Fig. 1) causes the phenyl ring to adopt a boat conformation and the nitro groups to twist ( $63$  and  $67^\circ$ ) with respect to the benzene nucleus.



$O(1)-N(1)-C(3)-C(2)$	67.1	$O(3)-N(2)-C(5)-C(6)$	-62.7
$O(2)-N(1)-C(3)-C(2)$	-109.8	$O(4)-N(2)-C(5)-C(6)$	114.1
$O(2)-N(1)-C(3)-C(4)$	66.8	$O(4)-N(2)-C(5)-C(4)$	-63.3
$O(1)-N(1)-C(3)-C(4)$	-116.3	$O(3)-N(2)-C(5)-C(4)$	119.8
$C(8)-C(7)-C(4)-C(5)$	-27.6	$C(10)-C(7)-C(4)-C(3)$	-89.6
$C(9)-C(7)-C(4)-C(3)$	32.5	$C(10)-C(7)-C(4)-C(5)$	94.5
$C(11)-O(5)-C(1)-C(6)$	5.0		
$C(3)-C(2)-C(1)-C(6)$	-2.4	$C(2)-C(1)-C(6)-C(5)$	1.9
$C(11)-C(6)-C(5)-C(4)$	2.0	$C(6)-C(5)-C(4)-C(3)$	-4.7
$C(5)-C(4)-C(3)-C(2)$	4.1	$C(4)-C(3)-C(2)-C(1)$	-0.8

Fig. 1. A view of  $C_{11}H_{14}N_2O_5$  and torsion angles.

## (3,4-DIHYDROXYPHENETHYL)TRIMETHYLAMMONIUM BROMIDE (CORYNEINE BROMIDE)



T.A. HAMOR and R.H. JONES, 1982. Acta Cryst., B38, 1007-1009.

 Monoclinic,  $P2_1/c$ ,  $a = 9.730$ ,  $b = 10.141$ ,  $c = 12.989$  Å,  $\beta = 101.55^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.041$  for 1638 reflexions.

In the coryneine cation (Fig. 1) the ethylammonium side chain is in the extended conformation with C-C-C-N<sup>+</sup> torsion angle  $179.8^\circ$ . The plane of these atoms is inclined at  $76^\circ$  to the plane of the phenyl ring. Two hydrogen bonds, between the phenolic groups and bromide ions, link the coryneine cations into chains parallel to c: O...Br<sup>-</sup> distances are 3.211 and 3.320 Å respectively. Other contact distances correspond to normal van der Waals interactions.

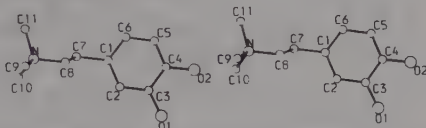
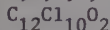


Fig. 1. Stereoscopic view of the coryneine cation in a direction perpendicular to the plane of the phenyl ring.

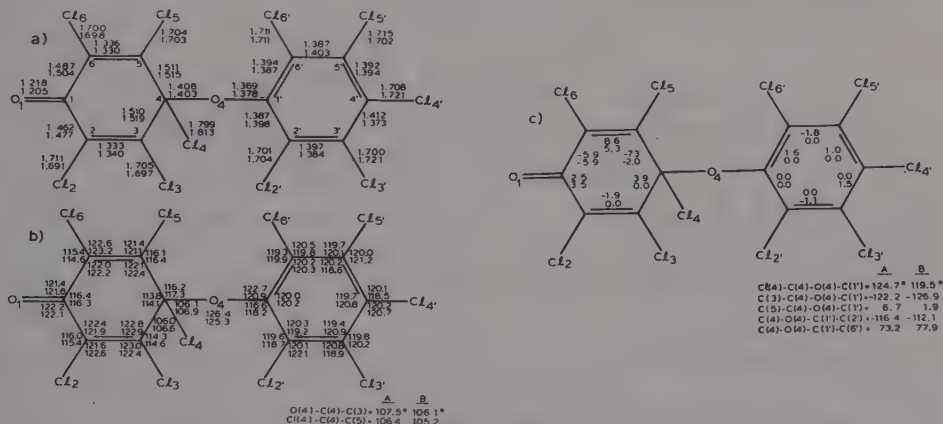
## 2,3,4,5,6-PENTACHLORO-4-(PENTACHLOROPHENOXY)-2,5-CYCLOHEXADIENONE



J.-A.B. CAMPBELL, M.L. DEINZER, T.L. MILLER, D.C. ROHRER and P.E. STRONG, 1982. J. Org. Chem., 47, 4968-4970.

 Monoclinic,  $Cc$ ,  $a = 14.178$ ,  $b = 8.6429$ ,  $c = 28.665$  Å,  $\beta = 98.869^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.062$  for 4813 reflexions.

There are two independent molecules in the asymmetric unit and both have the same conformation. The dihedral angles between the ring planes are  $68.1$  and  $66.5^\circ$  for the two molecules (Fig. 1).


 Fig. 1. Dimensions (bond lengths, angles and torsion angles) for the two  $C_{12}Cl_{10}O_2$  molecules.

## N-SALICYLIDENE-4-BROMOANILINE

 $C_{13}H_{10}BrNO$ 

S.V. LINDEMAN, V.E. SHKLOVER, Yu.T. STRUCHKOV, S.G. KRAVCHENY and V.M. POTAPOV, 1982. Cryst. Struct. Comm., 11, 49-52.

Orthorhombic,  $Pca2_1$ ,  $a = 6.173$ ,  $b = 6.916$ ,  $c = 26.197$  Å,  $Z = 4$ . Cu radiation,  $R = 0.050$  for 730 reflexions.

The molecule (Fig. 1) is non-planar; phenyl ring C(11)-C(16) is inclined at  $13.9^\circ$  to the plane of the C=N bond, and ring C(1)-C(6) makes a dihedral angle of  $42.8^\circ$  with the plane containing C=N.

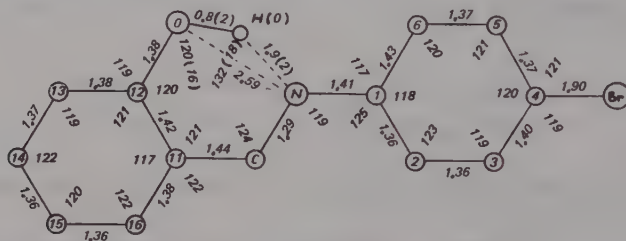


Fig. 1. Molecular dimensions for  $C_{13}H_{10}BrNO$ .

## N-SALICYLIDENE-4-CHLOROANILINE

 $C_{13}H_{10}ClNO$ 

S.V. LINDEMAN, V.E. SHKLOVER, Yu.T. STRUCHKOV, S.G. KRAVCHENY and V.M. POTAPOV, 1982. Cryst. Struct. Comm., 11, 43-47.

Monoclinic,  $P2_1/c$ ,  $a = 13.435$ ,  $b = 5.7902$ ,  $c = 14.722$  Å,  $\beta = 106.08^\circ$ ,  $D_m = 1.40$ ,  $Z = 4$ . Cu radiation,  $R = 0.062$  for 1448 reflexions.

The molecule (Fig. 1) is almost planar; the phenyl rings form interplanar angles of  $6^\circ$  with the plane of the C=N bond.

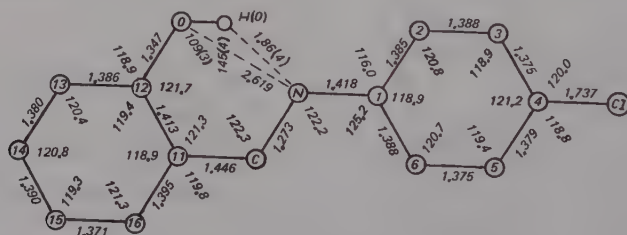


Fig. 1. Molecular dimensions for  $C_{13}H_{10}ClNO$ .

## 2,2'-DIHYDROXYBENZOPHENONE

 $C_{13}H_{10}O_3$ 

E.O. SCHLEMPER, 1982. Acta Cryst., B38, 1619-1622.

Monoclinic,  $P2_1/n$ ,  $a = 7.865$ ,  $b = 12.237$ ,  $c = 11.253$  Å,  $\beta = 109.73^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.033$  for 1278 reflexions.

Discrete molecules (Fig. 1) are linked by van der Waals forces and a weak hydrogen bond  $O(1)...O(3)$  3.024(2) Å. The planar six-membered rings are inclined at  $45.7^\circ$  to each other with  $H(C6)...H(C13)$  2.26 Å. Both OH groups are hydrogen bond donors to the carbonyl group. Some ring angles are: C(2)-C(1)-C(6)  $118.0^\circ$ , C(9)-C(8)-

C(13) 117.9, C(1)-C(6)-C(5) 121.2, C(8)-C(13)-C(12) 121.5°.

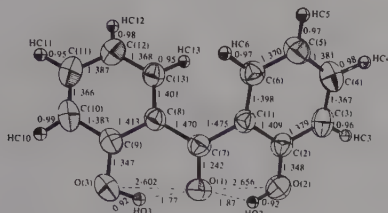


Fig. 1. Bond lengths in the 2,2'-dihydroxybiphenyl molecule.

2,2',4,4'-TETRAHYDROXYBENZOPHENONE  
C<sub>13</sub>H<sub>10</sub>O<sub>5</sub>

E.O. SCHLEMPER, 1982. *Acta Cryst.*, **B38**, 554-559.

Triclinic,  $P\bar{1}$ ,  $a = 9.950$ ,  $b = 12.479$ ,  $c = 9.334$  Å,  $\alpha = 98.80$ ,  $\beta = 93.22$ ,  $\gamma = 72.45^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.034$  for 2499 reflexions.

Each of the two symmetry-independent molecules has intramolecular hydrogen bonding between both ortho hydroxyl groups and the carbonyl oxygen with 0...0 2.516(2) to 2.716(2) Å (Fig. 1). The intermolecular hydrogen bonding scheme is different for the two molecules (Fig. 1). The dihedral angles between the two six-membered rings in each molecule are 41.7 and 43.5°. The benzene rings are slightly puckered with C(1) and C(4) on one side of the least-squares plane and C(2) and C(5) on the other. The crystal packing features almost parallel packing of the aromatic rings.

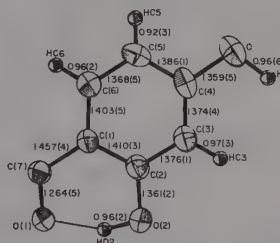


Fig. 1. Average values of chemically equivalent bond distances in C<sub>13</sub>H<sub>10</sub>O<sub>5</sub>.

N-BENZYLIDENE-p-HYDROXYANILINE  
C<sub>13</sub>H<sub>11</sub>NO

C<sub>6</sub>H<sub>5</sub>-CH=N-C<sub>6</sub>H<sub>4</sub>-OH

J. MEUNIER-PIRET, G. GERMAIN, J.P. DECLERCQ and M. VAN MEERSSCHE, 1982. *Bull. Soc. Chim. Belg.*, **91**, 89-90.

Monoclinic,  $P2_1/n$ ,  $a = 6.494$ ,  $b = 14.847$ ,  $c = 10.839$  Å,  $\beta = 91.51^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.041$  for 1155 reflexions.

In the molecule (Fig. 1) the N(8)=C(9) distance is 1.277(1) Å and the torsion angle C(5)-N(8)-C(9)-C(10) is 180.0°.



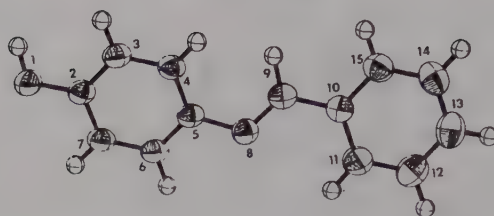
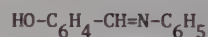


Fig. 1. The N-benzylidene-p-hydroxyaniline molecule.

N-(p-HYDROXYBENZYLIDENE)ANILINE

$C_{13}H_{11}NO$



J. MEUNIER-PIRET, G. GERMAIN, J.P. DECLERCQ and M. VAN MEERSSCHE, 1982. Bull. Soc. Chim. Belg., 91, 93-94.

Orthorhombic, Pbca,  $a = 20.031$ ,  $b = 10.861$ ,  $c = 9.450$  Å,  $Z = 8$ .  $R = 0.061$  for 895 reflexions.

The molecule is shown in Fig. 1. The  $N(8)=C(9)$  distance is  $1.274(3)$  Å and the torsion angle  $C5-N8-C9-C10$  is  $169.9^\circ$ .

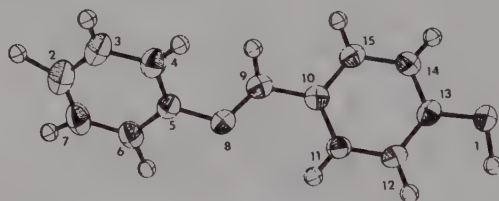


Fig. 1. A view of N-(p-hydroxybenzylidene)aniline.

N-(n-BUTYL)-N'-[(p-CHLOROPHENOXY)ACETYL]UREA

$C_{13}H_{17}ClN_2O_3$

S. VIJAY KUMAR and L.M. RAO, 1982. Acta Cryst., B38, 974-976.

Triclinic,  $P1$ ,  $a = 5.214$ ,  $b = 8.444$ ,  $c = 17.518$  Å,  $\alpha = 95.40$ ,  $\beta = 91.90$ ,  $\gamma = 113.99^\circ$ ,  $D_m = 1.332$ ,  $Z = 2$ . Cu radiation,  $R = 0.067$  for 837 reflexions (photographic data).

There is a short intramolecular N-H...O hydrogen bond ( $2.62$  Å) in the molecule (Fig. 1); the formation of this bond closes a part of the molecule to form a six-membered ring. A short intermolecular Cl...Cl distance of  $3.361$  Å is observed.

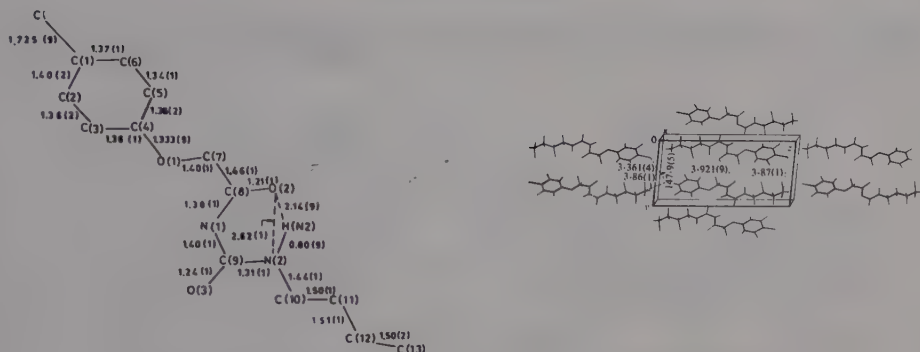
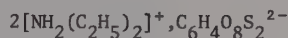
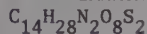


Fig. 1.  $C_{13}H_{17}ClN_2O_3$ : bond lengths (left) and the packing of the molecules viewed perpendicular to the bc plane (right).

## DIETHYLAMMONIUM 2,5-DIHYDROXY-1,4-BENZENEDISULPHONATE



X. SOLANS, F. PLANA and M. FONT-ALTABA, 1982. *Acta Cryst.*, B38, 651-653.

Monoclinic,  $P2_1/a$ ,  $a = 11.533$ ,  $b = 10.544$ ,  $c = 8.888$  Å,  $\beta = 114.67^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.043$  for 1122 reflexions.

The diethylammonium cation is linked to two anions (Fig. 1) lying on inversion centres, to one by a strong N-H...O hydrogen bond (2.787 Å) and to the other by two weak ones (2.999 and 3.152 Å). A strong O-H...O bond (2.694 Å) links the anions. The molecular geometry is normal.

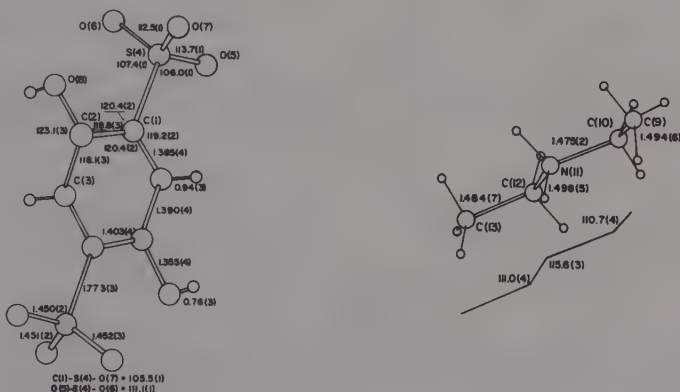
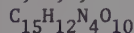


Fig. 1. Views of the ions in  $C_{14}H_{28}N_2O_8S_2$ .

2,2',6,6'-TETRANITRO-4,4'-ISOPROPYLIDENEDIPHENOL



J.L. WANG, C.P. TANG and Y.J. CHEN, 1982. *Acta Cryst.*, B38, 2286-2288.

Monoclinic, C2/c,  $a = 17.202$ ,  $b = 9.477$ ,  $c = 11.263$  Å,  $\beta = 107.0^\circ$ ,  $D_m = 1.535$ ,  $Z = 4$ . Cu radiation,  $R = 0.068$  for 1590 reflexions.

The molecule (Fig. 1) has  $C_2$  symmetry. The dihedral angle between the two phenyl-ring planes is  $63.9(5)^\circ$ . The -OH group forms an ordered intramolecular

hydrogen bond with one of the two neighbouring nitro groups with  $O(1) \cdots O(3)$  2.59(1) Å. The other nitro group is rotated through  $46.5(3)^\circ$  from coplanarity with the phenyl ring.

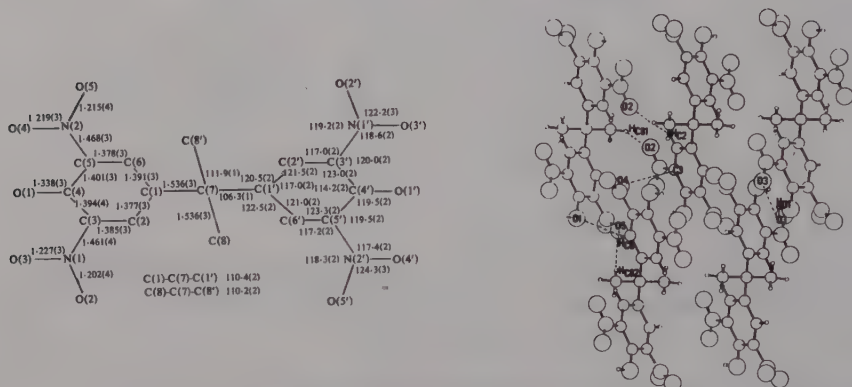


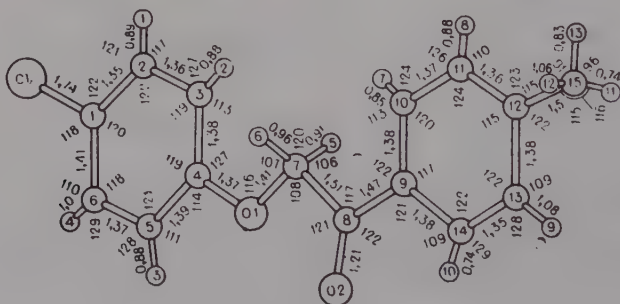
Fig. 1.  $C_{15}H_{12}N_4O_{10}$ : bond lengths and angles in the molecule and the molecular packing arrangement viewed along  $b$ ; short contacts ( $<3.0$  Å) are indicated by broken lines.

#### 4'-CHLOROPHENOXYACETYL-4-METHYLBENZENE $C_{15}H_{13}ClO_2$

T. ATABAEV, Yu.V. GATILOV, N.V. PODBEREZSKAYA, A. ASHIROV and S.V. BORISOV, 1982. Zh. Strukt. Khim., 23-5, 179-181 [J. Struct. Chem., 23, 819-821].

Orthorhombic,  $P2_1ab$ ,  $a = 21.466$ ,  $b = 11.770$ ,  $c = 5.186$  Å,  $Z = 4$ . Mo radiation,  $R = 0.054$  for 1115 reflexions.

The molecular geometry is shown in Fig. 1 ( $\sigma = 0.007$ - $0.010$  Å and  $<0.5^\circ$  for nonhydrogen atoms). The aromatic rings are planar within experimental error, the angle between the mean planes being  $10.0^\circ$ .



The molecular geometry ( $\sigma = 0.01 \text{ \AA}$  and  $0.4\text{--}0.6^\circ$  for non-hydrogen atoms) is shown in Fig. 1. Both aromatic rings are planar within experimental error, the angle between mean planes being  $18.3^\circ$ .

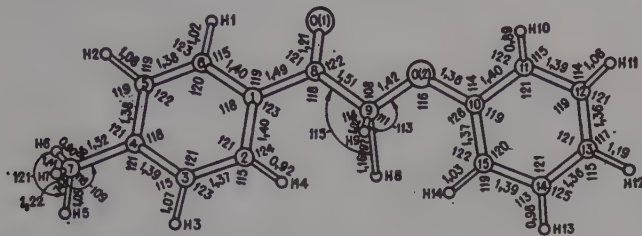


Fig. 1. The 1-phenoxyacetyl-4-methylbenzene molecule.

( $\pm$ )-2-(4-(4-CHLOROPHENOXYMETHYL)PHENOXY)PROPIONIC ACID

$C_{16}H_{15}ClO_4$

G. SMITH, C.H.L. KENNARD, W.L. DUAX and D.C. SWENSON, 1982. *Aust. J. Chem.*, **35**, 2151-2155.

Triclinic,  $P\bar{1}$ ,  $a = 9.345$ ,  $b = 12.602$ ,  $c = 6.741 \text{ \AA}$ ,  $\alpha = 101.37$ ,  $\beta = 97.42$ ,  $\gamma = 101.75^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.084$  for 2172 reflexions.

The acid molecules (Fig. 1) form centrosymmetric hydrogen-bonded dimers ( $O\cdots O$  2.619(4)  $\text{\AA}$ ). The oxopropionic acid side chain has a synplanar-synplanar (carbonyl) conformation, while the nearly planar p-chlorophenoxyethyl substituent in the p-position has a synclinal orientation relative to the primary phenoxy residue ( $C(3)C(4)-C(81)O(71)$   $82.0(4)^\circ$ ).

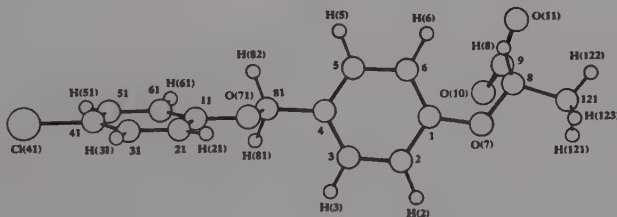


Fig. 1. Molecular conformation of ( $\pm$ )-2-(4-(4-chlorophenoxyethyl)phenoxy)propionic acid.

(E)-1-(2-BROMO-4,5-DIMETHOXYPHENYL)-2-(3,4-DIMETHOXYPHENYL)ETHYLENE

$C_{18}H_{19}BrO_4$

J.M. ARRIETA, E. LETE, E. DOMINGUEZ, G. GERMAIN, J.P. DECLERCQ and J.M. AMIGO, 1982. *Acta Cryst.*, **B38**, 3155-3157.

Monoclinic,  $C2/c$ ,  $a = 28.65$ ,  $b = 9.340$ ,  $c = 12.795 \text{ \AA}$ ,  $\beta = 95.05^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.047$  for 1384 reflexions.

The molecule (Fig. 1) is in the trans form and is almost planar (dihedral angle of  $9^\circ$  between the two phenyl planes).

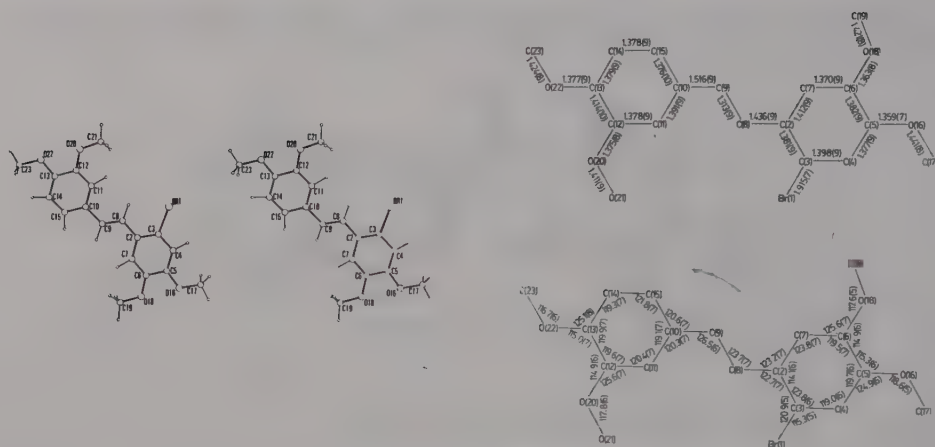


Fig. 1. A stereoview of  $C_{18}H_{19}BrO_4$  and dimensions.

2,3'-DINITRO-4,5'-DIMETHYL-5,4'-DIETHOXYBIPHENYL  
 $C_{18}H_{20}N_2O_6$

M.M. NAFISSI-V, D.P. KOHARSKI, M.S. PUAR and A.T. McPHAIL, 1982. J. Org. Chem., **47**, 3345-3348.

Monoclinic,  $P2_1/c$ ,  $a = 7.878$ ,  $b = 8.204$ ,  $c = 27.843$  Å,  $\beta = 91.36^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.076$  for 923 reflexions.

Relief from severe steric overcrowding involving adjacent phenyl ring substituents is gained by various combinations of significant exocyclic bond angle deformation with out-of-plane twisting and bending. Thus, the dihedral angles between the plane through C(1)-C(6) and those through C(2),N(7),O(8),O(9), and C(1')-C(6') are  $34.8$  and  $46.7^\circ$ , respectively (Fig. 1). In addition, the highly significant enlargements of the C(1)-C(2)-N(7) and C(2)-C(1)-C(1') angles over the smaller C(3)-C(2)-N(7) and C(6)-C(1)-C(1') angles serve to alleviate nonbonded interactions between the C(2)-nitro and C(1)-phenyl ring B substituents. At C(5), the ethoxy substituent adopts the usual preferred conformation, with C(12) lying close to the phenyl ring plane. Repulsive nonbonded interactions between the C(12) methylene group and the phenyl ring A hydrogen at C(6) are relieved through increase in both the C(6)-C(5)-O(11) and C(5)-O(11)-C(12) angles and concomitant decrease in the C(4)-C(5)-O(11) angle. The dihedral angle between the planes through phenyl ring B carbon atoms and that through C(3'),N(7'),O(8'), and O(9') is  $49.6^\circ$ . Moreover, owing to the fact that the C(4')-ethoxy substituent is sandwiched between the C(3')-nitro and C(5')-methyl substituents, C(11') is forced out of the phenyl ring B plane by  $1.111$  Å to relieve severe steric overcrowding; the associated C(3')-C(4')-O(10')-C(11') torsion angle is  $-107.1^\circ$ .

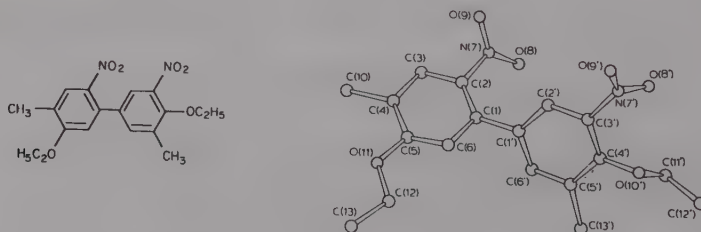


Fig. 1. The  $C_{18}H_{20}N_2O_6$  molecule.

N-{2-[2-METHYL-3-(p-NITROBENZYL)OXY-1-ISOTHIUREIDO]ETHYL}-p-TOLUENESULFONAMIDE  
 $C_{18}H_{22}N_4O_5S_2$

Z. URBĄŃCZYK-LIPKOWSKA, J.W. KRAJEWSKI, P. GLUZIŃSKI and K. STADNICKA, 1982. Acta Cryst., B38, 971-973.

Triclinic,  $P\bar{1}$ ,  $a = 8.127$ ,  $b = 7.128$ ,  $c = 18.040$  Å,  $\alpha = 86.50$ ,  $\beta = 97.13$ ,  $\gamma = 94.30^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.043$  for 2973 reflexions.

The molecule (Fig. 1) has an open-chain conformation with an N-H...O hydrogen bond involving the amidine system and the benzyloxy O atom. Steric effects of substituents at the C=N bond, the conjugation in the amidine system and probably also the packing forces cause a curved conformation of the chain. A short van der Waals N...N contact of 2.947 Å is found in the molecule.

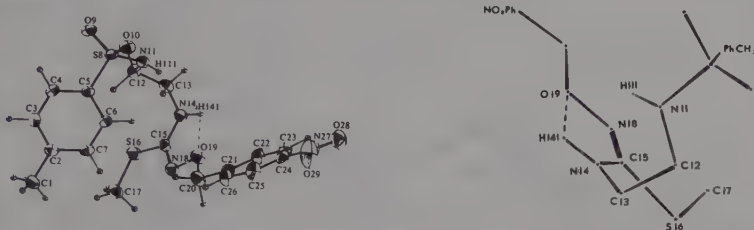


Fig. 1.  $C_{18}H_{22}N_4O_5S_2$ : perspective views of the molecule.

SYNEPHRINE MONOHYDROGENPHOSPHATE MONOHYDRATE

$C_{18}H_{29}N_2O_8P, H_2O$

$2C_9H_{14}NO_2^+, HPO_4^{2-}, H_2O$

J.K. DATTAGUPTA, E.F. MEYER and B.P. MUKHOPADHYAY, 1982. Acta Cryst., B38, 2830-2834.

Monoclinic,  $P2_1/n$ ,  $a = 12.108$ ,  $b = 8.402$ ,  $c = 21.549$  Å,  $\beta = 99.67^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.056$  for 2562 reflexions.

The two molecules in the asymmetric unit have different conformations, one of them being closely similar to other sympathomimetic amines (Fig. 1). Bond lengths and angles in molecule A are generally as expected, but in molecule B C(7) and O(2) may be disordered, with consequential effects on molecular geometry. The crystal structure is stabilised by a three-dimensional network of O-H...O and N-H...O hydrogen bonds, with O...O 2.542 to 2.825 and N...O 2.732 to 2.884 Å.

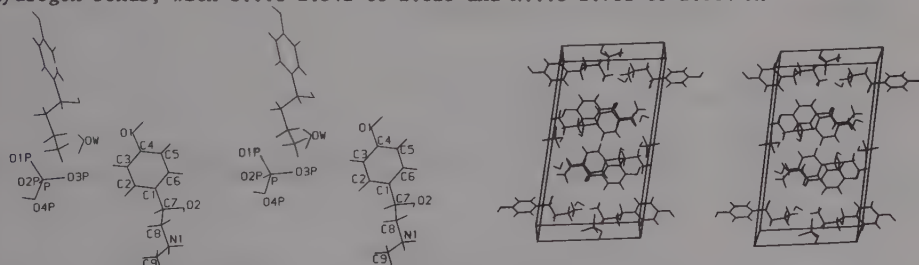


Fig. 1.  $C_{18}H_{29}N_2O_8P$ : stereoviews of the asymmetric unit and of the crystal structure.

2,2'-(PHENOXYMETHYLENE) BIS(4-CHLORO-6-NITROPHENOL)

$C_{19}H_{12}Cl_2N_2O_7$  (I)

2,2'-(BENZYLOXYMETHYLENE) BIS(4-CHLORO-6-NITROPHENOL) TRIETHYLAMINE SOLVATE

$C_{20}H_{14}Cl_2N_2O_7, C_6H_{15}N$  (II)



D.G. HAY and M.F. MACKAY, 1982. *Aust. J. Chem.*, **35**, 341-349.

I. Monoclinic,  $P2_1/n$ ,  $a = 8.616$ ,  $b = 16.214$ ,  $c = 13.727$  Å,  $\beta = 92.13^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.061$  for 1761 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 9.454$ ,  $b = 11.911$ ,  $c = 25.392$  Å,  $\beta = 109.96^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.063$  for 1230 reflexions.

In I (Fig. 1) the methylenebisphenol moiety adopts the twist conformation ( $C(1)C(7)-C(1')(C(2')) -71.6(4)^\circ$ ,  $C(1')C(7)-C(1)C(6) -61.1(4)^\circ$ ) with the ortho hydroxy groups at C(2) and C(2') on the same side of the  $C(1)C(7)C(1')$  plane and distal and proximal respectively. The dihedral angle between rings A and A' (see below) is  $80.1(5)^\circ$  and the molecule assumes an overall T-shape. In II (Fig. 1) the central moiety has only small deviations from the idealized butterfly form ( $C(1)C(7)-C(1')C(2')$   $85.6(7)^\circ$ ,  $C(1')C(7)-C(1)C(6)$   $88.0(8)^\circ$  and again the ortho hydroxy groups are on the same side of the  $C(1)C(7)C(1')$  plane. The dihedral angle between rings A and A' is  $118.2(8)^\circ$ . In both molecules the nitro groups are rotated from coplanarity with their phenyl rings ( $1.8(3)$ ,  $13.4(3)^\circ$  (I);  $25.3(6)$ ,  $33.5(6)^\circ$  (II)).

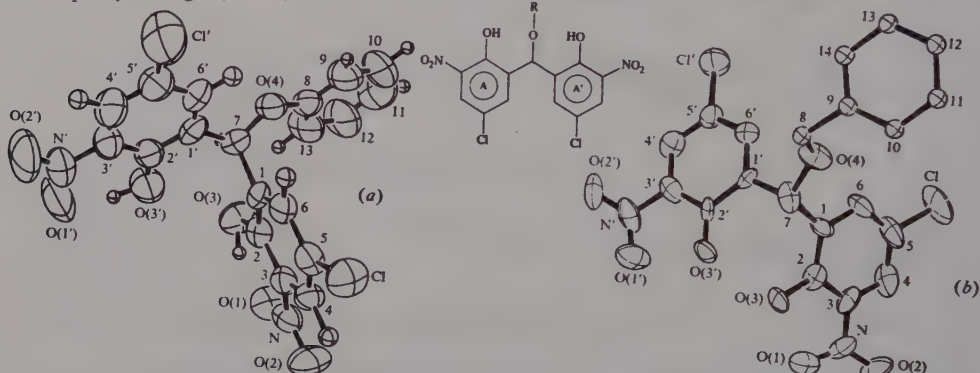


Fig. 1. Perspective views of I (a) and II (b).

# BIS(SALICYLIDENE)-o-PHENYLENEDIIMINE

$C_{20}H_{16}N_2O_2$

C. SUBRAHMANYAM, M. SESHASAYEE and G. ARAVAMUDAN, 1982. *Cryst. Struct. Comm.*, **11**, 1719-1723.

Monoclinic,  $P2_1/c$ ,  $a = 5.981$ ,  $b = 16.579$ ,  $c = 16.347$  Å,  $\beta = 91.275^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.07$  for 1032 reflexions.

In this molecule (Fig. 1) main bond lengths are  $N(1)-C(1)$   $1.426(11)$ ,  $N(1)-C(7)$   $1.319(11)$ ,  $N(2)-C(2)$   $1.405(11)$ ,  $N(2)-C(14)$   $1.285(11)$ ,  $O(1)-C(13)$   $1.354(11)$ ,  $O(2)-C(16)$   $1.349(11)$  Å.

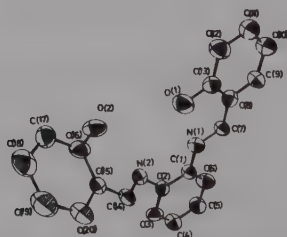


Fig. 1. A view of  $C_{20}H_{16}N_2O_2$ .

## 1,7-BIS(4-HYDROXY-3-METHOXYPHENYL)-1,6-HEPTADIEN-3,5-DIONE

 $C_{21}H_{20}O_6$ 

H.H. TØNNESEN, J. KARLSEN and A. MOSTAD, 1982. Acta Chem. Scand., B36, 475-479.

Monoclinic,  $P2_1/n$ ,  $a = 20.028$ ,  $b = 7.073$ ,  $c = 12.609$  Å,  $\beta = 94.94^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.055$  for 3331 reflexions (at 121 K).

The molecule has three substituted planar groups connected by the two double bonds C(7)-C(8) and C(12)-C(13) (Fig. 1). The two terminal groups are identical but a rotation of  $-162^\circ$  about the C(11)-C(12) bond destroys the inherent symmetry of the molecule. Atoms O(3) and O(4) of the enol ring are engaged in inter- as well as intramolecular hydrogen bonding (Fig. 1). The best model to fit the data is the one with the hydrogen atom statistically distributed between O(3) and O(4), and not localised on one O atom.

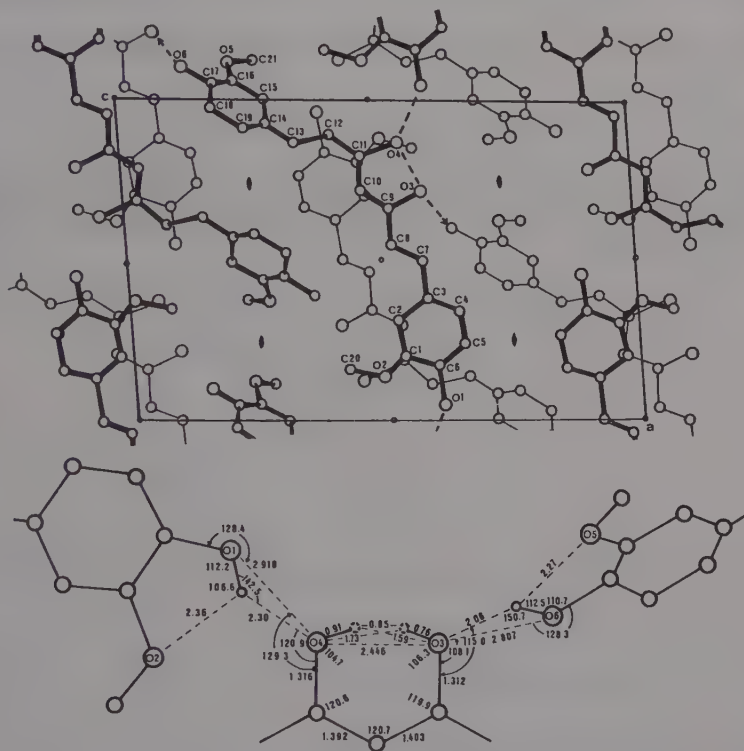


Fig. 1. Molecular packing in crystals of  $C_{21}H_{20}O_6$  (top), and geometry and hydrogen bonding in the enol ring (bottom).

## 2,2'-METHYLENEBIS(4-CHLORO-6-ISOPROPYL-5-METHYLPHENOL)

 $C_{21}H_{26}Cl_2O_2$ 

S. RANTSORDAS and M. PERRIN, 1982. Acta Cryst., B38, 1871-1873.

Monoclinic,  $P2_1/n$ ,  $a = 12.915$ ,  $b = 9.157$ ,  $c = 17.566$  Å,  $\beta = 105.22^\circ$ ,  $D_m = 1.29$ ,  $Z = 4$ . Cu radiation,  $R = 0.043$  for 2676 reflexions.

The molecule has the following interplane angles (Fig. 1): 2/4  $107.1(1)$ , 1/2  $94.3(3)$ , 4/5  $93.1(3)$ , 2/3  $100.3$ , 3/4  $102.3^\circ$ . The C(2)-C(21)-C(12) angle is  $112.1(3)^\circ$ . There are no short intermolecular contacts.

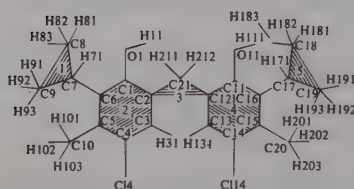


Fig. 1. Atomic and plane numbering schemes in  $C_{21}H_{26}Cl_2O_2$ .

1-[p-(2-BROMOETHOXY)PHENYL]-2-CHLORO-1,2-DIPHENYLETHYLENE  
 $C_{22}H_{18}BrClO$

G. PRÉCIGOUX, M. HOSPITAL, F. LEROY, A. DELBARRE and B.P. ROQUES, 1982. Acta Cryst., B38, 312-315.

Monoclinic, Cc,  $a = 12.317$ ,  $b = 15.238$ ,  $c = 9.982$  Å,  $\beta = 94.32^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.056$  for 2046 reflexions.

In the molecule (Fig. 1) the dihedral angle between phenyl rings on the same carbon is  $74^\circ$ . The bromoethoxy chain is not in a trans conformation and the Br-C-C-O dihedral angle is  $71.8^\circ$ . The molecules are held together in the crystal by van der Waals forces.

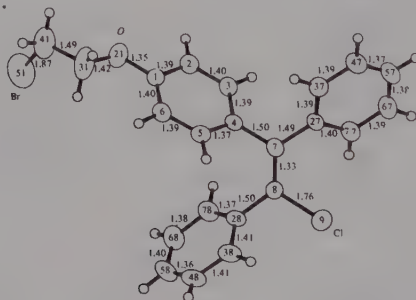


Fig. 1. Bond lengths in the  $C_{22}H_{18}BrClO$  molecule.

(RR)-BIS[1-(2,4,6-TRIMETHYLPHENYL)ETHYL] ETHER  
 $C_{22}H_{30}O$  (I)

(RS)-BIS[1-(2,4,6-TRIMETHYLPHENYL)ETHYL] ETHER  
 $C_{22}H_{30}O$  (II)

C. FOCES-FOCES, F.H. CANO and S. GARCÍA-BLANCO, 1982. Acta Cryst., B38, 1957-1962.

I. Monoclinic,  $P2_1/c$ ,  $a = 8.0827$ ,  $b = 31.6784$ ,  $c = 7.7624$  Å,  $\beta = 103.092^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.053$  for 2217 reflexions.

II. Orthorhombic,  $Pc2_1n$ ,  $a = 11.8337$ ,  $b = 10.3267$ ,  $c = 15.5781$  Å,  $Z = 4$ . Cu radiation,  $R = 0.067$  for 1193 reflexions.

The analysis shows which molecule is RR and which is RS (Fig. 1). Main bond length differences are (RR first): O(2)-C(3) 1.426(4) and 1.366(10), C(3)-C(5) 1.521(4) and 1.496(13), C(3)-C(31) 1.519(4) and 1.563(11) Å. At C(1), O(2)-C(1)-C(4) is  $106.4$  and  $113.2^\circ$ , O(2)-C(1)-C(11)  $113.9$  and  $108.9^\circ$ . The angles between the rings are  $30.3$  and  $40.3^\circ$  with the ring planes about  $48^\circ$  from the O-C-C plane.

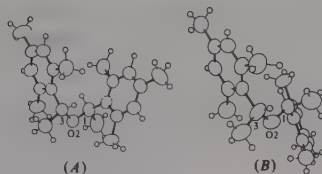


Fig. 1. The relative configurations of the RR (A) and RS (B) molecules of  $C_{22}H_{30}O$ .

1-(p-METHOXYPHENYL)-trans-1,2-DIPHENYLBUT-1-ENE  
 $C_{23}H_{22}O$

S.D. CUTBUSH, S. NEIDLE, A.B. FOSTER and F. LECLERCQ, 1982. Acta Cryst., B38, 1024-1027.

Triclinic, P1,  $a = 9.644$ ,  $b = 10.0001$ ,  $c = 10.4999$  Å,  $\alpha = 78.77$ ,  $\beta = 67.53$ ,  $\gamma = 74.91^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.0586$  for 2294 reflexions.

There are two independent molecules with very similar molecular geometry in the asymmetric unit (Fig. 1). The propeller conformation adopted is seen in related structures; the angles between the planar phenyl rings are in the range  $54-62^\circ$ .

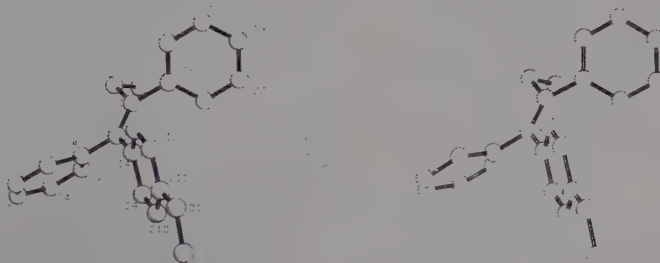


Fig. 1.  $C_{23}H_{22}O$ : the two independent molecules in the asymmetric unit, viewed from an identical projection.

(E)-3,4-BIS(p-METHOXYPHENYL)-2,2,5,5-TETRAMETHYL-3-HEXENE  
 $C_{24}H_{32}O_2$

T. PILATI and M. SIMONETTA, 1982. Acta Cryst., B38, 1649-1652.

Monoclinic,  $P2_1/c$ ,  $a = 10.792$ ,  $b = 11.930$ ,  $c = 8.326$  Å,  $\beta = 93.97^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.076$  for 1306 reflexions.

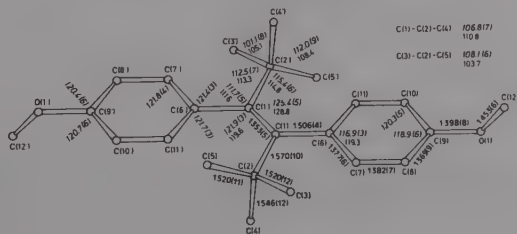
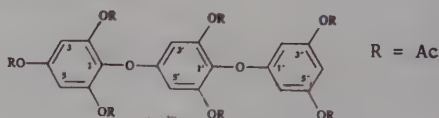


Fig. 1. One of the  $C_{24}H_{32}O_2$  molecules projected onto the C(1), C(4), C(6) plane. (Values shown without e.s.d.'s refer to theoretical results.)

The centrosymmetric molecule (Fig. 1) lies at a centre of symmetry with two statistically disordered orientations. Dimensions are normal.

2,2',4,6,6'-PENTAACETOXY-1'-(3,5-DIACETOXYPHENOXY)BIPHENYL ETHER

$C_{32}H_{28}O_{16}$



R.P. GREGSON and J.J. DALY, 1982. Aust. J. Chem., 35, 649-657.

Triclinic,  $P\bar{1}$ ,  $a = 10.128$ ,  $b = 10.487$ ,  $c = 17.526$  Å,  $\alpha = 94.2$ ,  $\beta = 100.2$ ,  $\gamma = 115.6^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.046$  for 2022 reflexions.

The structure was confirmed as that shown above with normal values for the molecular dimensions.

BIS(4'-p-NONYLOXYBENZYLIDENE)-1,4-PHENYLENEDIAMINE

$C_{38}H_{52}N_2O_2$

$[C_3H_2OC(H)C_6H_4N(CH_2)_8CH_3]_2$

L.M. KAMENCHUK, E.A. KOSTERIN, Yu.V. NEKRASOV and G.N. TISHCHENKO, 1982.

Kristallografiya, 27, 501-505 [Sov. Phys. Crystallogr., 27, 304-306].

Monoclinic,  $P2_1/b$ ,  $a = 36.893$ ,  $b = 6.173$ ,  $c = 7.380$  Å,  $\gamma = 90.27^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.065$  for 1698 reflexions.

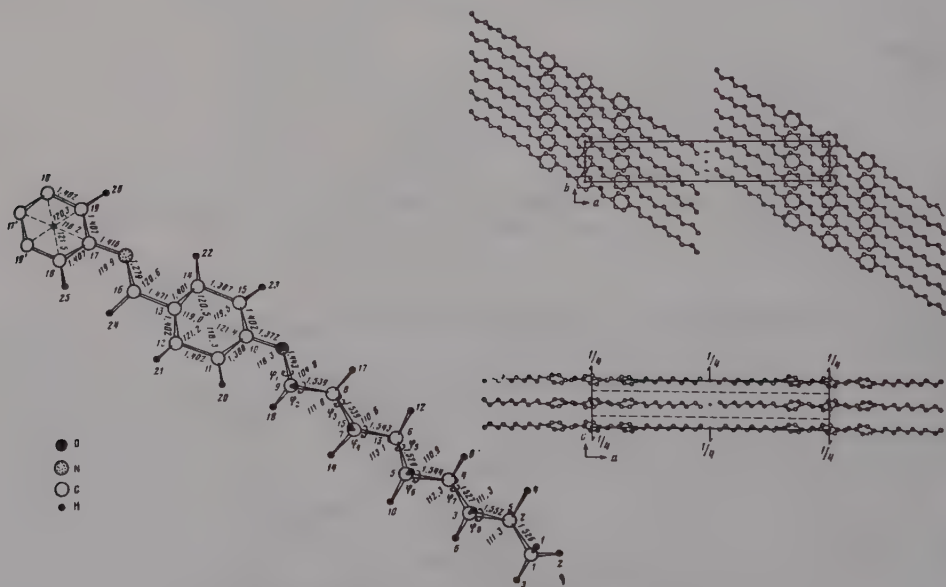
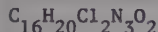


Fig. 1. Asymmetric half of  $C_{38}H_{52}N_2O_2$  showing bond lengths (Å) and angles ( $^\circ$ ) (left), and structure projections on  $ab$  (top right) and  $ac$  (bottom right) planes.

The centrosymmetric molecule, half of which is shown in Fig. 1, is 43.44 Å in length and has an elongated rod-shaped form. All C atoms in the terminal aliphatic  $C_8H_{19}$  chains, and the O atom are in trans positions while the chains themselves are at an angle of  $2.6^\circ$  to the axis of the molecule. The angle between the benzylidene and phenyl rings is  $4.72^\circ$ . In the crystal the molecules are packed in layers parallel to the ab plane; the layers are spaced  $c/2$  apart (Fig. 1). The long axes of the molecules make an angle of  $31.1^\circ$  with the a axis.

TETRAETHYLAMMONIUM 2,3-DICHLORO-5,6-DICYANO-p-BENZO-QUINONE



G. ZANOTTI, A. DEL PRA and R. BOZIO, 1982. Acta Cryst., B38, 1225-1229.

Monoclinic,  $P2_1/n$ ,  $a = 12.493$ ,  $b = 20.114$ ,  $c = 6.996$  Å,  $\beta = 98.79^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.062$  for 2427 reflexions.

In the crystal structure the quinoid ring of the anion (Fig. 1) is planar, with the substituents significantly displaced from its mean plane. The anions are stacked with different intervals in the crystal, the interplanar spacing being 2.906 and 3.626 Å. The shorter value suggests that the present structure could be represented as the packing of dimeric anions.

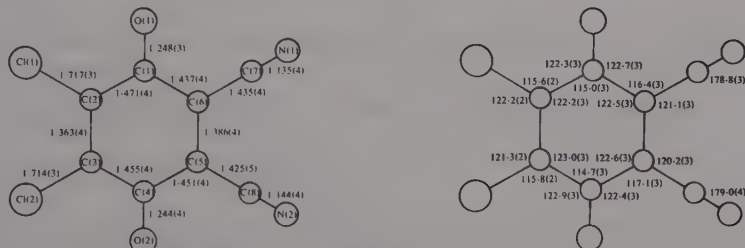
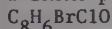


Fig. 1.  $C_{16}H_{20}Cl_2N_3O_2$ : bond distances and angles in the anion.

$\omega$ -BROMO-p-CHLOROACETOPHENONE



S.B. GUPTA and K.N. PRASAD, 1982. Z. Krist., 158, 159-163.

Orthorhombic,  $P2_12_12_1$ ,  $a = 9.649$ ,  $b = 21.871$ ,  $c = 4.174$  Å,  $D_m = 1.75$ ,  $Z = 4$ . Cu radiation,  $R = 0.084$  for Weissenberg data.

A view of the structure in projection is shown in Fig. 1. Molecular dimensions are in accord with expected values.

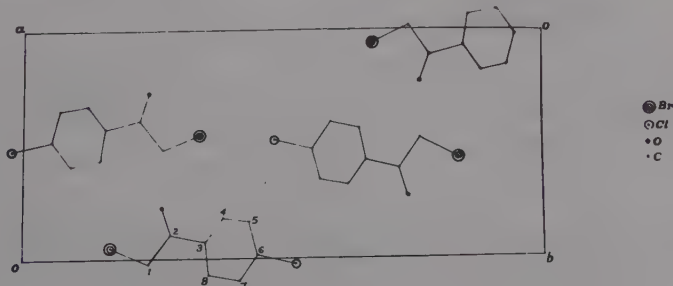


Fig. 1. The projection along c of  $C_8H_6BrClO$ .



## 2H,2'H-OCTAFLUOROBIPHENYL



J. BOWEN JONES and D.S. BROWN, 1982. *Acta Cryst.*, B38, 317-318.

Orthorhombic,  $Pbca$ ,  $a = 21.710$ ,  $b = 7.545$ ,  $c = 12.79$  Å,  $Z = 8$ . Cu radiation,  $R = 0.045$  for 1078 reflexions.

In the molecule (Fig. 1) the phenyl rings are each planar, with an inter-ring bond length and dihedral angle of 1.491 Å and 54.7° respectively. Intra-ring C-C bond lengths are 1.367-1.400 Å (mean 1.380 Å), C-F bond lengths are 1.330-1.353 Å (mean 1.346 Å).

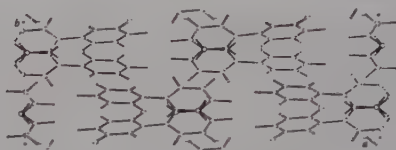
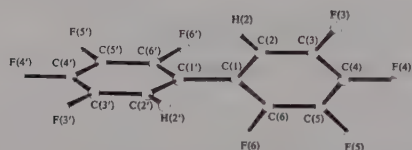
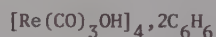
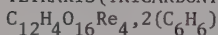


Fig. 1. A view of the  $C_{12}H_2F_8$  molecule and the projection of the contents of the unit cell down c.

TETRAKIS (TRICARBONYL( $\mu_3$ -HYDROXO)RHENIUM) BENZENE SOLVATE

B. NUBER, F. OBERDORFER and M ZIEGLER, 1981. *Acta Cryst.*, B37, 2062-2064.

Cubic, Pn3m,  $a = 11.343 \text{ \AA}$ ,  $D_m = 3.0$ ,  $Z = 2$ . Mo radiation,  $R = 0.029$  for 225 reflexions.

The structure (Fig. 1) consists of discrete tetramers with four Re and four O atoms occupying the corners of a distorted cube. The benzene rings, two per tetrameric unit, are perpendicular to the four triad axes with the centres of the rings on these axes at a distance 3.285 Å from the O atoms of the four  $\mu_3$ -OH groups. The benzene rings probably use both faces as acceptors for OH... $\pi$  hydrogen bonding.

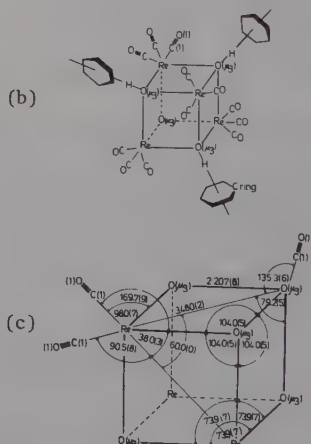
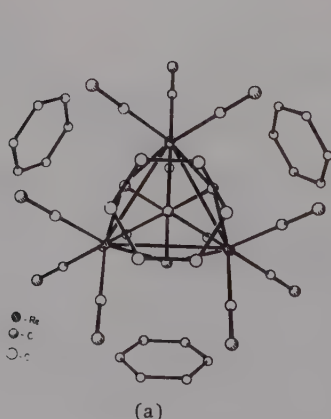
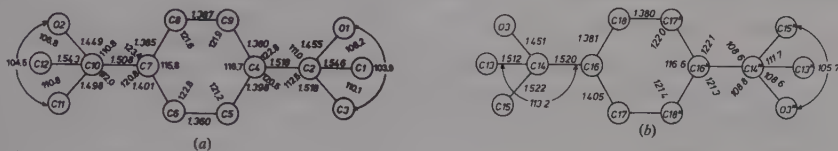


Fig. 1.  $\text{C}_{12}\text{H}_4\text{O}_{16}\text{Re}_4 \cdot 2(\text{C}_6\text{H}_6)$ : (a) view of the cubane-like structure along one of the threefold axes, (b) schematic view in which the fourth benzene ring has been omitted for clarity, and (c) distances and angles in the distorted cube.

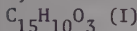
## 2,2'-(p-PHENYLENE)DI-2-PROPANOL

T. KORITSÁNSZKY and G. MENCZEL, 1982. *Acta Cryst.*, **B38**, 1617-1619.Triclinic,  $\bar{P}1$ ,  $a = 10.269$ ,  $b = 14.369$ ,  $c = 6.276$  Å,  $\alpha = 98.21$ ,  $\beta = 96.62$ ,  $\gamma = 70.15^\circ$ ,  $D_m = 1.10$ ,  $Z = 3$ . Mo radiation,  $R = 0.068$  for 1211 reflexions.

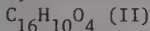
The asymmetric unit contains one molecule in general position and one half molecule at a centre of symmetry. Molecular geometry is shown in Figure 1; average C-C distances are 1.387 (aromatic) and 1.520 Å (aliphatic). The cis oxygens in molecule 1 are about 0.2 Å from the ring plane while O(3) in molecule 2 is displaced 0.8 Å. The oxygen atoms of three adjacent molecules are linked by hydrogen bonds ( $0 \dots 0$  2.757-2.848 Å) producing spirals parallel to  $c$  and sheets parallel to  $(110)$ .

Fig. 1. The two independent  $\text{C}_{12}\text{H}_{18}\text{O}_2$  molecules.

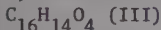
## 1,3-DIPHENYLPROPANE-1,2,3-TRIONE



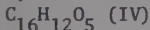
## 1,4-DIPHENYLBUTANE-1,2,3,4-TETRAONE



## 2,4-DIHYDROXY-2,5-DIPHENYLFURAN-3(2H)-ONE



## 3,3-DIHYDROXY-1,4-DIPHENYLBUTANE-1,2,4-TRIONE

R.L. BEDDOES, J.R. CANNON, M. HELLER, O.S. MILLS, V.A. PATRICK, M.B. RUBIN and A.H. WHITE, 1982. *Aust. J. Chem.*, **35**, 543-556.I. Triclinic,  $\bar{P}1$ ,  $a = 9.756$ ,  $b = 8.813$ ,  $c = 8.303$  Å,  $\alpha = 68.56$ ,  $\beta = 67.53$ ,  $\gamma = 89.11^\circ$ ,  $D_m = 1.30$ ,  $Z = 2$ . Mo radiation,  $R = 0.036$  for 942 reflexions.II. Monoclinic,  $P2_1/c$ ,  $a = 10.641$ ,  $b = 8.381$ ,  $c = 14.710$  Å,  $\beta = 91.70^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.029$  for 979 reflexions.III. Monoclinic,  $P2_1/c$ ,  $a = 5.741$ ,  $b = 9.276$ ,  $c = 24.31$ ,  $\beta = 106.26^\circ$ ,  $D_m = 1.42$ ,  $Z = 4$ . Mo radiation,  $R = 0.067$  for 941 reflexions.IV. Triclinic,  $\bar{P}1$ ,  $a = 13.08$ ,  $b = 12.43$ ,  $c = 8.895$ ,  $\alpha = 82.54$ ,  $\beta = 81.87$ ,  $\gamma = 78.61^\circ$ ,  $D_m = 1.34$ ,  $Z = 4$ . Mo radiation,  $R = 0.045$  for 1661 reflexions.

In I (Fig. 1) the two phenyl groups are approximately coplanar with the adjacent carbonyl group (dihedral angles  $15.5^\circ$  for phenyl 1 and  $4.2^\circ$  for phenyl 2). Bond lengths and angles generally have expected values but the  $\text{O}(1)\text{C}(1)\text{C}(2)$  angle ( $114.8(3)^\circ$ ) is unusually small. The structure of II (Fig. 1) is similar to I with dihedral angles between the phenyl groups and adjacent carbonyls of  $6.3^\circ$  and  $16.8^\circ$  for phenyl 1 and phenyl 2 respectively. The O-C-C angles in the outer carbonyls are even further diminished to  $112.2(2)$  and  $113.4(2)^\circ$  ( $\text{O}(1)\text{C}(1)\text{C}(2)$  and  $\text{C}(3)\text{C}(4)\text{O}(4)$  respectively). In III (Fig. 1) the molecules are linked across inversion centres by O-H...O hydrogen bonds ( $0 \dots \text{H}$  1.87(7), 2.22(6) Å). The  $\text{C}(2,3,4,21), \text{O}(4,3)$  moiety is approximately planar and the furan ring is in an envelope conformation. The dihedral angle between phenyl 2 and the furan ring is  $7.7^\circ$ . In IV (Fig. 1) as in I and II the phenyl rings and adjacent carbonyls are nearly coplanar (dihedral angles  $2.8$  and  $8.1^\circ$  for phenyl 1 and 2 respectively). Two short hydrogen bonds are found between  $\text{O}(2')$

(molecule 1) and H(2) (molecule 1) (1.95(5) Å) and between O(1) (molecule 2) and H(2') (molecule 1) (2.06(8) Å).

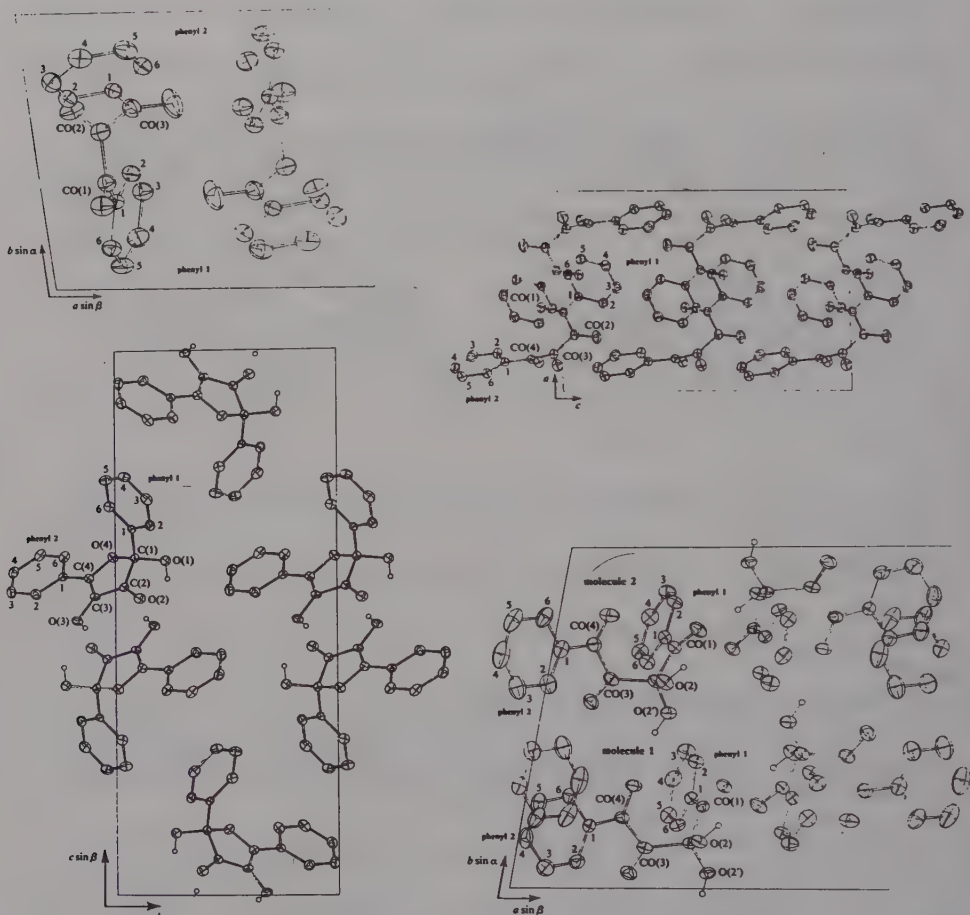


Fig. 1. Views of the unit cell contents of the diphenyl compounds (I) top left, (II) top right, (III) lower left, (IV) lower right.

(RS,SR) -3-BROMO-2-METHYL-1,3-DIPHENYLPROPAN-1-ONE  
 $C_{16}H_{15}BrO$  (I)

(RR,SS) -2-(BROMOPHENYLMETHYL) -3,3-DIMETHYL-1-PHENYLBUTAN-1-ONE  
 $C_{19}H_{21}BrO$  (II)

(1R,2R,4R,5S/1S,2S,4S,5R) -2,4-DIBENZOYL-1,5-DIBROMO-1,5-DIPHENYLPENTANE  
 $C_{31}H_{26}Br_2O_2$  (III)

(RRR,SSS) -REL-(2R,3R,5R) -3-BENZOYL-2,3,4,5-TETRAHYDRO-4,4-DIMETHYL-2,5-DIPHENYL-INDENO[1,2-b]PYRAN  
 $C_{33}H_{28}O_2$  (IV)

H. QUAST, B. MÜLLER, K. PETERS and H.G. von SCHNERING, 1982. *Chem. Ber.*, **115**, 1525-1546.

I. Orthorhombic,  $Aba2$ ,  $a = 9.805$ ,  $b = 28.28$ ,  $c = 9.905$  Å,  $Z = 8$ . Mo radiation,  $R = 0.094$  for 652 reflexions.

II. Orthorhombic,  $Pna2_1$ ,  $a = 14.839$ ,  $b = 19.210$ ,  $c = 5.933$  Å,  $Z = 4$ . Mo radiation,  $R = 0.073$  for 1151 reflexions.

III. Monoclinic,  $C2/c$ ,  $a = 21.516$ ,  $b = 10.262$ ,  $c = 24.684$  Å,  $\beta = 102.22^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.054$  for 3444 reflexions.

IV. Triclinic,  $P\bar{1}$ ,  $a = 12.493$ ,  $b = 16.430$ ,  $c = 6.484$  Å,  $\alpha = 100.73$ ,  $\beta = 98.78$ ,  $\gamma = 101.46^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.072$  for 3810 reflexions.

In I (Fig. 1) the ideal staggered configuration of the propane chain is distorted by Br-C(20) repulsion which opens the C(20)C(2)-C(3) Br torsion angle to  $75^\circ$ . In II (Fig. 1) a similar distortion is caused by the phenyl-*t*-butyl repulsion (C(30)C(3)-C(2)C(20)  $67^\circ$ ). In III (Fig. 1) the zig-zag arrangement of the chain gives a minimum of gauche interactions and an antiperiplanar placing of the largest substituents at bonds C(1)-C(2), C(2)-C(3) and C(3)-C(4), but not however at C(4)-C(5). In IV (Fig. 1) the pyran ring substituents are trans. The indene moiety and O(1) are coplanar within 0.03 Å; C(11) lies 0.20 Å below this plane.

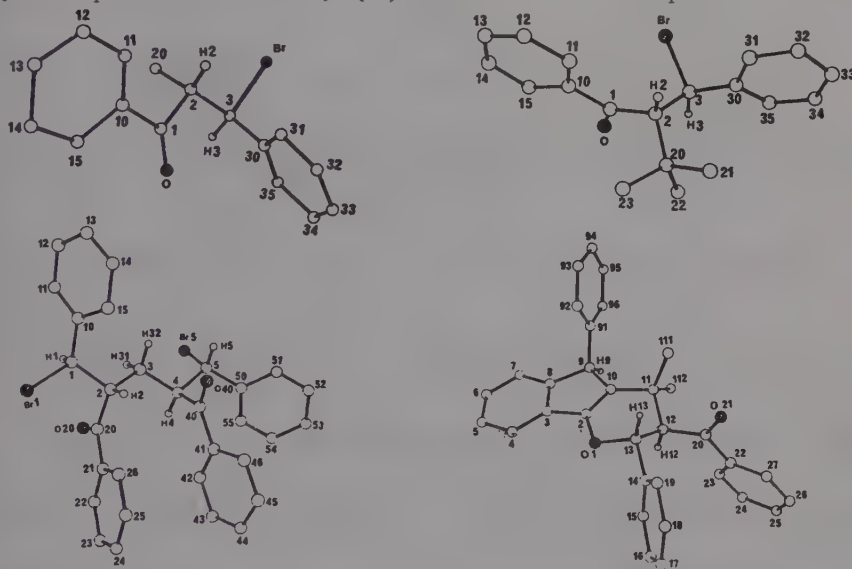


Fig. 1. Views of  $C_{16}H_{15}BrO$  (top left),  $C_{19}H_{21}BrO$  (top right),  $C_{31}H_{26}Br_2O$  (bottom left), and  $C_{33}H_{28}O_2$  (bottom right).

TRIPHENYLCHLOROMETHANE  
 $C_{19}H_{15}Cl$

$(C_6H_5)_3CCl$

A. DUNAND and R. GERDIL, 1982. *Acta Cryst.*, **B38**, 570-575.

Trigonal,  $P\bar{3}$ ,  $a = 13.998$ ,  $c = 13.202$  Å,  $D_m = 1.26$ ,  $Z = 6$ . Mo radiation,  $R = 0.038$  for 905 reflexions.

Each of the three, independent, propeller-shaped molecules has its C-Cl bond coincident with a crystallographic threefold axis. The molecules are associated in

pairs and form two distinct  $\text{Ph}_3\text{C}-\text{Cl}\dots\text{Cl}-\text{CPh}_3$  head-to-head arrangements characterised by long C-Cl bonds (1.874(6), 1.847(6), 1.843(6) Å) and short Cl...Cl contacts (3.210 Å). The C-C-Cl angles are in the range  $106-107^\circ$  with C-C-C angles  $111.9-112.7^\circ$ .

(-)-PHENYL  $\alpha$ -(1-PHENYLPROPYLIMINO)BENZYL KETONE

$\text{C}_{23}\text{H}_{21}\text{NO}$

I. FONSECA, S. MARTÍNEZ-CARRERA and S. GARCÍA-BLANCO, 1982. Acta Cryst., B38, 2735-2738.

Monoclinic,  $P2_1$ ,  $a = 19.188$ ,  $b = 9.2573$ ,  $c = 10.739$  Å,  $\beta = 100.190^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.037$  for 5535 reflexions.

The enantiomer studied has an R configuration. The two independent molecules are non-planar and have similar conformations (Fig. 1). Bond lengths and angles are also similar and are generally as expected. The C(7)-C(8) bond (1.524(2) (A) and 1.517(3) Å (B)) is nearer to a  $\text{C}(\text{sp}^3)-\text{C}(\text{sp}^3)$  bond (1.536 Å) than the expected  $\text{C}(\text{sp}^2)-\text{C}(\text{sp}^2)$  one (1.482 Å). Intermolecular contacts correspond to van der Waals interactions.

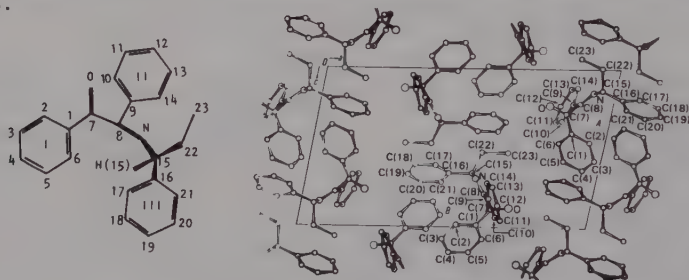


Fig. 1.  $\text{C}_{23}\text{H}_{21}\text{NO}$ : structural formula showing absolute configuration and the packing diagram as viewed along the  $b$  axis.

PHENYL  $\alpha$ [( $\alpha$ -PHENYLBENZYL)IMINO]BENZYL KETONE

$\text{C}_{27}\text{H}_{21}\text{NO}$

I. FONSECA, S. MARTÍNEZ-CARRERA and S. GARCÍA-BLANCO, 1982. Acta Cryst., B38, 3121-3123.

Monoclinic,  $P2_1/n$ ,  $a = 15.347$ ,  $b = 9.0877$ ,  $c = 15.205$  Å,  $\beta = 100.850^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.051$  for 2789 reflexions.

A view of the molecule is in Fig. 1. Comparison with other imines shows that introduction of the phenyl ring at C(15) does not affect the bond distances or angles but does alter the conformation of the central C-C=N-C skeleton.

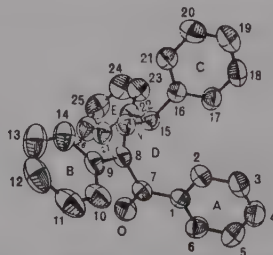
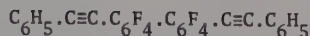


Fig. 1. A view of  $\text{C}_{27}\text{H}_{21}\text{NO}$ .



## OCTAFLUORO-4,4'-BIS(PHENYLETHYNYL)BIPHENYL

 $C_{28}H_{10}F_8$ 

N. GOODHAND and T.A. HAMOR, 1982. *Acta Cryst.*, **B38**, 1342-1345.

Monoclinic,  $C2/c$ ,  $a = 23.86$ ,  $b = 6.22$ ,  $c = 14.98$  Å,  $\beta = 100.01^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.042$  for 1387 reflexions.

In the crystal structure the molecules possess exact  $C_2$  symmetry, with the crystallographic twofold axis perpendicular to and bisecting the central bond (Fig. 1). The acetylenic triple-bond length is 1.187 Å. The inter-ring bond of the octafluorobiphenyl system is 1.477 Å and the inter-ring dihedral angle  $56.7^\circ$ .

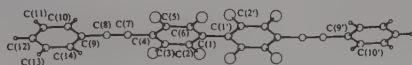


Fig. 1.  $C_{28}H_{10}F_8$ : a view of the molecule parallel to  $b$ .

1,2-BIS(2,4,6-TRI-*t*-BUTYLPHENYL)ETHANE $C_{38}H_{62}$ 

W. WINTER, T. BUTTERS, A. RIEKER and Y. BUTSUGAN, 1982. *Z. Naturforsch.*, **37b**, 855-862.

Monoclinic,  $C2/c$ ,  $a = 10.923$ ,  $b = 17.910$ ,  $c = 17.395$  Å,  $\beta = 91.87^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.073$  for 3853 reflexions (at 173 K).

The molecule (Fig. 1) has crystallographic  $C_2$  symmetry and on account of steric repulsions, the central ethane bond is stretched to 1.567(4) Å. Close packing of the molecules in layers parallel to the ac-plane leads to boat deformations of the benzene rings.

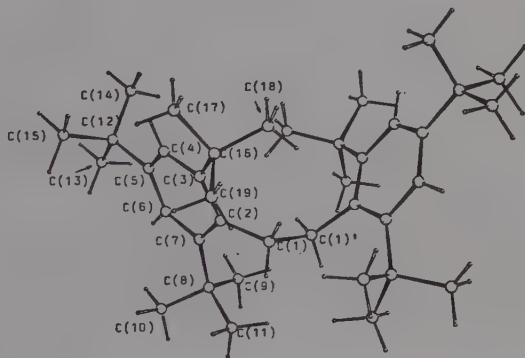


Fig. 1. Perspective view of  $C_{38}H_{62}$ .

## 1,1,1,3,3,3-HEXAPHENYLPROPANE

 $C_{39}H_{32}$ 

W. WINTER, A. MOOSMAYER and A. RIEKER, 1982. *Z. Naturforsch.*, **37b**, 1623-1632.

Monoclinic,  $C2/c$ ,  $a = 9.943$ ,  $b = 19.054$ ,  $c = 14.708$  Å,  $\beta = 101.80^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.046$  for 2625 reflexions.

The molecule (Fig. 1) in the monoclinic form has  $C_2$ -symmetry. Compared to the triclinic form (1) there are no significant differences in bond lengths or angles in



the central part of the molecule but there are major differences of 10-16° in the torsion angles which describe the slope of the phenyl propellers. Principal bond lengths are: C(1)-C(2) 1.566, C(2)-C(3) 1.544, C(2)-C(9) 1.546, C(2)-C(15) 1.564 Å.

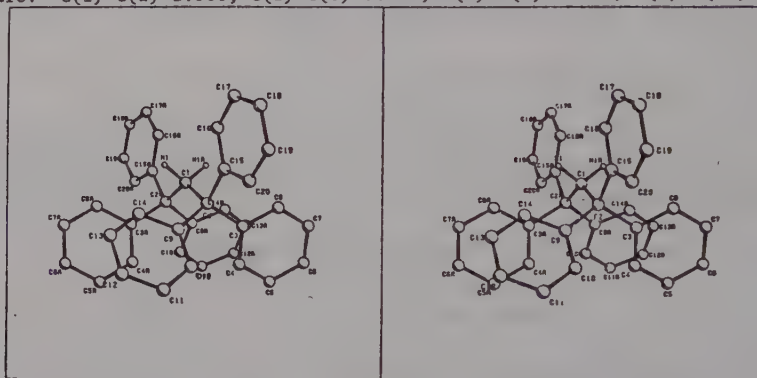
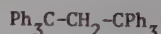


Fig. 1. Stereoview of  $C_{39}H_{32}$ .

1. Following Report.

1,1,1,3,3,3-HEXAPHENYLPROPANE (Triclinic form)  
 $C_{39}H_{32}$



J.F. BLOUNT, A. GUTIERREZ and K. MISLOW, 1982. J. Org. Chem., 47, 4359-4361.

Triclinic,  $P\bar{1}$ ,  $a = 9.267$ ,  $b = 10.654$ ,  $c = 15.976$  Å,  $\alpha = 88.13$ ,  $\beta = 74.53$ ,  $\gamma = 67.31^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.040$  for 3117 reflexions.

The conformation adopted by this crowded structure is shown in Fig. 1 in which the molecule has approximate  $C_2$  symmetry. The central C-CH<sub>2</sub>-C bond angle is widened to  $128.0(2)^\circ$ .

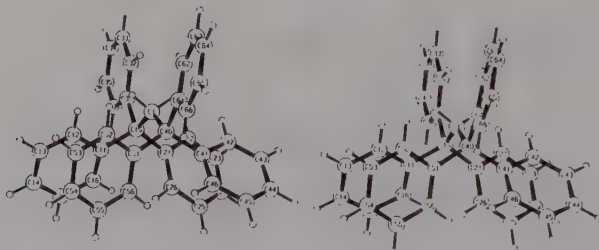


Fig. 1. A stereoview of  $C_{39}H_{32}$ .

3-METHYLENE-trans-1,2-CYCLOPROPANEDICARBOXYLIC ACID (FEIST'S ACID)  
 $C_6H_6O_4$

N. RAMASUBBU and K. VENKATESAN, 1982. Acta Cryst., B38, 976-978.

Triclinic,  $P\bar{1}$ ,  $a = 4.842$ ,  $b = 7.607$ ,  $c = 9.168$  Å,  $\alpha = 98.41$ ,  $\beta = 99.89$ ,  $\gamma = 77.74^\circ$ ,  $D_m = 1.45$ ,  $Z = 2$ . Mo radiation,  $R = 0.038$  for 723 reflexions.

The bond lengths in the cyclopropane ring are C(1)-C(2) 1.461, C(2)-C(3) 1.546 and C(3)-C(1) 1.464 Å. The molecules (Fig. 1) are arranged in the crystal in infinite chains with intermolecular O-H...O hydrogen bonds with O...O 2.637 and 2.675 Å almost parallel to  $b$ . For previous work see (1).

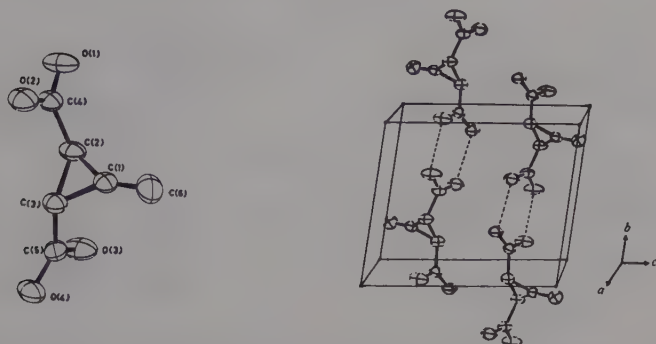


Fig. 1. Feist's Acid: perspective view of the molecule (left) and the molecular packing viewed down *a* (right).

1. Structure Reports, 18, 694; 20, 547.

METHYL 3,5-DICHLORO-1,4-DIHYDROXY-2-METHYLCYCLOPENT-2-ENE-1-CARBOXYLATE  
 $C_8H_{10}Cl_2O_4$

G.M. STRUNZ and P.S. WHITE, 1982. *Canad. J. Chem.*, 60, 2062-2064.

Monoclinic,  $P2_1/c$ ,  $a = 7.352$ ,  $b = 8.868$ ,  $c = 16.300$ ,  $\beta = 96.49^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.057$  for 1706 reflexions.

The analysis established the structure and stereochemistry of this intermediate in cryptosporiopsin synthesis to be as shown in Fig. 1.

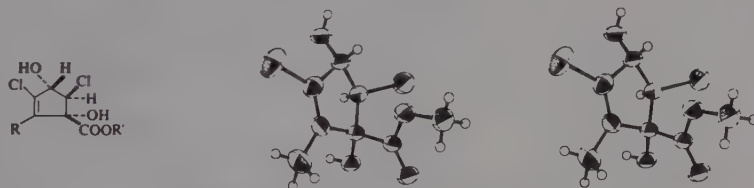


Fig. 1. The  $C_8H_{10}Cl_2O_4$  molecule and a stereoview.

2-CYCLOPENTYLPROPANEDIOIC ACID

$C_8H_{12}O_4$

J.L. WANG, C.P. TANG and C.C. CHIEN, 1982. *Cryst. Struct. Comm.*, 11, 1981-1988.

Triclinic,  $P\bar{1}$ ,  $a = 4.877$ ,  $b = 8.184$ ,  $c = 11.287$  Å,  $\alpha = 88.43^\circ$ ,  $\beta = 86.27^\circ$ ,  $\gamma = 85.32^\circ$ ,  $D_m = 1.275$ ,  $Z = 2$ . Cu radiation,  $R = 0.098$  for 1217 reflexions.

The carboxyl groups are linked by O-H...O hydrogen bonds about inversion centres, leading to infinite chains in the *c*-direction (Fig. 1). The dihedral angle between the two carboxyl group planes is  $83.2^\circ$ .

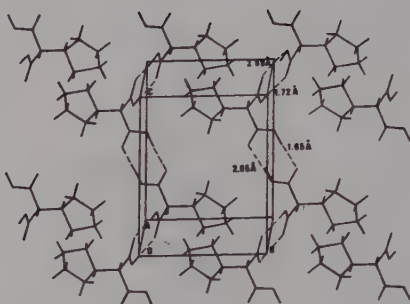
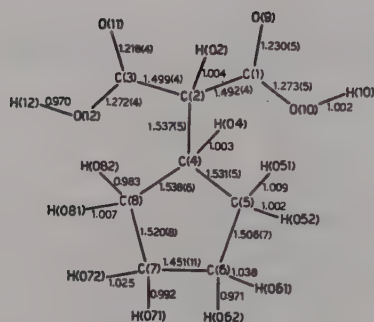


Fig. 1. Bond lengths and hydrogen bonding in  $C_8H_{12}O_4$ .

trans-2-PIVALOYLCYCLOPROPANECARBOXYLIC ACID



R. ROQUES, F. CRASNIER, J.P. DECLERCQ, G. GERMAIN, H. COUSSE and G. MOUZIN, 1982. *Acta Cryst.*, B38, 1375-1377.

Monoclinic,  $P2_1/n$ ,  $a = 5.888$ ,  $b = 16.237$ ,  $c = 10.252$  Å,  $\beta = 96.56^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.047$  for 865 reflexions.

In the molecule (Fig. 1) the cyclopropane ring has bond lengths 1.526, 1.478 and 1.493 Å. The molecules are linked into centrosymmetric dimers in the crystal by O-H...O hydrogen bonds (O...O 2.623(6) Å).

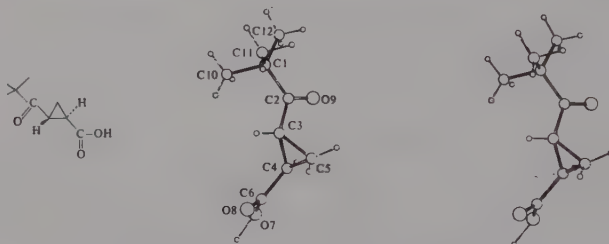
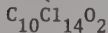


Fig. 1.  $C_9H_{14}O_3$ : a stereoscopic view of the molecule.

BIS(HEPTACHLORO-CYCLOPENT-2-ENYL)-PEROXIDE



S. GÄB, W.V. TURNER, F. KORTE and L. BORN, 1982. *J. Org. Chem.*, 47, 173-175.

Triclinic,  $P\bar{1}$ ,  $a = 6.255$ ,  $b = 8.413$ ,  $c = 10.872$ ,  $\alpha = 85.55^\circ$ ,  $\beta = 96.28^\circ$ ,  $\gamma = 113.4^\circ$ ,  $Z = 1$ .  $R = 0.045$  for 1017 reflexions.

The chlorine-sensitized oxidation of hexachlorocyclopentadiene produces a perchlorinated peroxide identified by X-ray analysis as that in Fig. 1. The compound crystallises with a centrosymmetric molecule and is thus the meso isomer. The C-C bonds about C5 are markedly long (1.57 Å) probably resulting from Cl1-Cl1 interactions. The cyclopentene ring has an envelope configuration with C5 0.51 Å out of the mean plane through the other four C-atoms.

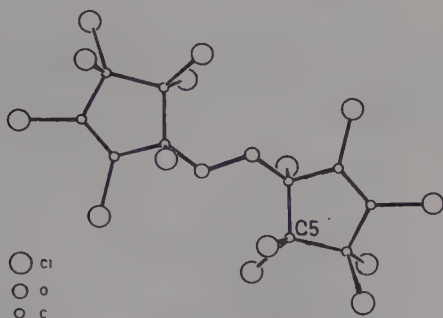


Fig. 1. A molecule of  $C_{10}Cl_{14}O_2$ .

ETHYL 1,2,2-TRICYANO-3,3-DIMETHYLCYCLOPROPANE-1-CARBOXYLATE  
 $C_{11}H_{11}N_3O_2$  (I)

ETHYL 2',3',3'-TRICYANOCYCLOHEXANESPIROCYCLOPROPANE-2'-CARBOXYLATE  
 $C_{14}H_{15}N_3O_2$  (II)

N. RAMASUBBU, J. RAJARAM and K. VENKATESAN, 1982. *Acta Cryst.*, **B38**, 196-199.

I. Orthorhombic,  $Pna2_1$ ,  $a = 14.662$ ,  $b = 10.492$ ,  $c = 7.375$  Å,  $D_m = 1.25$ ,  $Z = 4$ .  
 Mo radiation,  $R = 0.040$  for 782 reflexions.

II. Monoclinic,  $P2_1/a$ ,  $a = 7.886$ ,  $b = 22.011$ ,  $c = 8.100$  Å,  $\beta = 103.12^\circ$ ,  $D_m = 1.23$ ,  
 $Z = 4$ . Mo radiation,  $R = 0.042$  for 1400 reflexions.

In molecules I and II (Fig. 1) the bond C(1)-C(4), in each case flanked by electron-withdrawing groups, is significantly longer (1.542 Å in I and 1.545 Å in II) than the 1.510 Å average bond length found in the unsubstituted cyclopropane molecule (1). The observed bond lengths C(2)-C(3) (1.520 Å in I and 1.528 Å in II) and C(3)-C(1) (1.531 Å in I and 1.534 Å in II) are also significantly longer than 1.510 Å. The results are compared with those for similar related compounds.

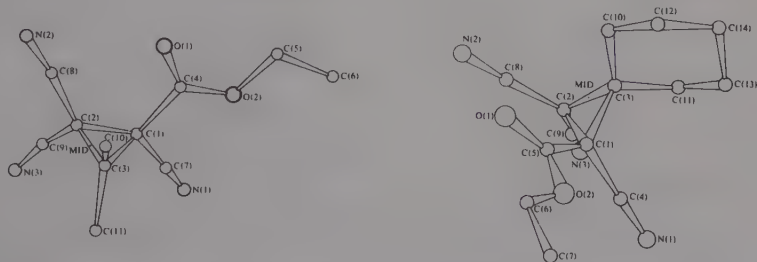


Fig. 1. Perspective views of  $C_{11}H_{11}N_3O_2$  (I) (left) and  $C_{14}H_{15}N_3O_2$  (II) (right).

# 1. Structure Reports, 29, 490.

r-1-METHOXY-1-METHYLTHIO-c-4-t-5-DIMETHYL-2,2,3,3-TETRACYANOCYCLOPENTANE  
 $C_{13}H_{14}N_4SO$

J.D. SCHAGEN, 1982. *Cryst. Struct. Comm.*, **11**, 1651-1653.

Monoclinic,  $P2_1/a$ ,  $a = 13.664$ ,  $b = 7.933$ ,  $c = 13.702$  Å,  $\beta = 96.80^\circ$ ,  $Z = 4$ . Cu  
 radiation,  $R = 0.042$  for 2167 reflexions.

The cyclopentane ring (Fig. 1) is an envelope conformation, with C(1), C(2), C(4) and C(5) in one plane (maximum deviation 0.012 Å) and C(3) 0.70 Å out of this plane. The two methyl groups are trans relative to each other. Four of the ring bonds are long; there is a clear correlation between the bond lengths and the number and the bulkiness of the substituents on the atoms forming the bonds.

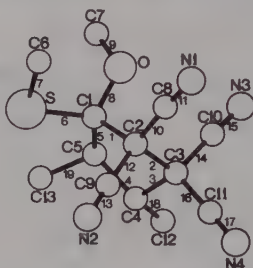


Fig. 1. A view of  $C_{13}H_{14}N_4SO$ .

1-ETHOXYCARBONYL-3-PHENYL-1-CYCLOBUTANECARBOXYLIC ACID



V. PRELOG, J. THIX and T. SRIKRISHNAN, 1982. *Helv. Chim. Acta*, **65**, 2622-2644.

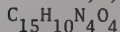
Triclinic,  $P\bar{1}$ ,  $a = 11.042$ ,  $b = 5.933$ ,  $c = 11.371$  Å,  $\alpha = 100.83$ ,  $\beta = 114.24$ ,  $\gamma = 91.67^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.066$  for 2219 reflexions.

The cyclobutane ring (Fig. 1) is puckered, with ring torsion angles  $\pm 19^\circ$  and a fold of  $27^\circ$ . Molecules are linked to form centrosymmetric dimers through the carboxyl groups.



Fig. 1. A stereoview of  $C_{14}H_{16}O_4$ .

ETHYL 1,2,2-TRICYANO-3-(4-NITROPHENYL)CYCLOPROPANE-1-CARBOXYLATE



N. RAMASUBBU and K. VENKATESAN, 1982. *Acta Cryst.*, **B38**, 1372-1375.

Monoclinic,  $P2_1/c$ ,  $a = 10.694$ ,  $b = 11.743$ ,  $c = 12.658$  Å,  $\beta = 113.10^\circ$ ,  $D_m = 1.38$ ,  $Z = 4$ . Mo radiation,  $R = 0.054$  for 1398 reflexions.

In the molecule (Fig. 1) bond lengths in the cyclopropane ring are 1.557, 1.504 and 1.461 Å and bond angles are  $61.9$ ,  $58.4$  and  $59.7^\circ$  respectively. Bond lengths in the ethoxy-carbonyl group, which is in an extended conformation, and in the cyano groups, which are non-linear C-C-N  $177.3$ ,  $177.6$  and  $178.4(4)^\circ$ , are normal. The packing of the molecules in the crystal indicates reasonably strong acid-base interactions.

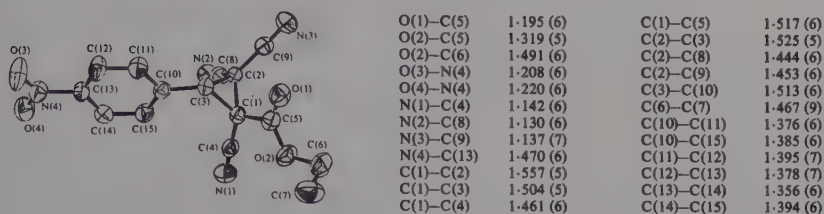


Fig. 1.  $C_{15}H_{10}N_4O_4$ : the molecule viewed down  $b$  (left) and bond lengths.

1,1-DICYANO-2,2,3-TRIMETHOXY-4-(trans)-PHENYLCYCLOBUTANE



J.M.M. SMITS, 1982. Cryst. Struct. Comm., 11, 715-719.

Triclinic,  $P\bar{1}$ ,  $a = 7.922$ ,  $b = 8.044$ ,  $c = 11.859$  Å,  $\alpha = 96.17^\circ$ ,  $\beta = 102.01^\circ$ ,  $\gamma = 98.91^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.037$  for 2027 reflexions.

The four-membered ring is folded, with a dihedral angle of  $37.62^\circ$  between the planes of C(1),C(2),C(3) and C(1),C(3),C(4) (Fig. 1). The phenyl group at C(4) and methoxyl at C(3) are trans.

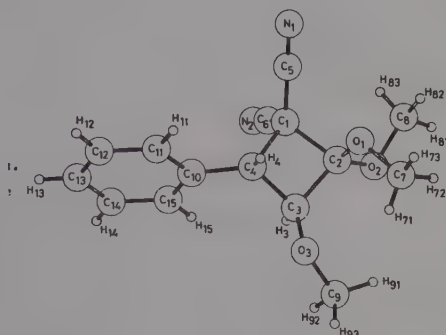
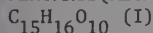
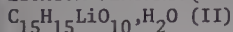


Fig. 1. A view of  $C_{15}H_{16}N_2O_3$ .

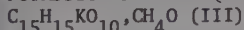
PENTAKIS(METHOXYCARBONYL) CYCLOPENTADIENE



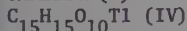
LITHIUM PENTAKIS(METHOXYCARBONYL) CYCLOPENTADIENIDE MONOHYDRATE



POTASSIUM PENTAKIS(METHOXYCARBONYL) CYCLOPENTADIENIDE METHANOL SOLVATE



THALLIUM(I) PENTAKIS(METHOXYCARBONYL) CYCLOPENTADIENIDE



M.I. BRUCE, J.K. WALTON, M.L. WILLIAMS, S.R. HALL, B.W. SKELTON and A.H. WHITE, 1982. J. Chem. Soc. Dalton, 2209-2220.

I. Triclinic,  $P\bar{1}$ ,  $a = 10.944$ ,  $b = 10.891$ ,  $c = 7.743$  Å,  $\alpha = 91.32^\circ$ ,  $\beta = 101.28^\circ$ ,  $\gamma = 113.91^\circ$ ,  $D_m = 1.44$ ,  $Z = 2$ . Mo radiation,  $R = 0.037$  for 1484 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 7.990$ ,  $b = 21.890$ ,  $c = 11.170$  Å,  $\beta = 109.99^\circ$ ,  $D_m = 1.37$ ,  $Z = 4$ . Mo radiation,  $R = 0.044$  for 1469 reflexions.



III. Orthorhombic,  $Pcab$ ,  $a = 13.52$ ,  $b = 14.793$ ,  $c = 19.751$  Å,  $D_m = 1.44$ ,  $Z = 8$ . Mo radiation,  $R = 0.042$  for 1695 reflexions.

IV. Monoclinic,  $P2_1/n$ ,  $a = 11.098$ ,  $b = 17.726$ ,  $c = 8.644$  Å,  $\beta = 96.47^\circ$ ,  $D_m = 2.18$ ,  $Z = 4$ . Mo radiation,  $R = 0.038$  for 3159 reflexions.

The molecule of I (Fig. 1) has a hydroxyfulvene type structure with the acid H atom bridging two of the carbonyl O atoms from adjacent COOMe groups ( $H...O(41,51)$  1.12(4), 1.32(4) Å,  $O...H...O$  171(3)°). Carboxylates 2, 4 and 5 lie parallel to the plane of the central  $C_5H_5$  ring while 1, 3 are normal to this ring. In the ring the  $C(4)-C(5)$  distance is 1.453(5) while the other distances range from 1.396(3) to 1.410(5) Å. There is extensive delocalisation over eight bonds in the molecule. In II, III and IV, salts of I, the integrity of the  $C_5H_5$  ring is preserved while the disposition of the carboxylates relative to this ring varies from coplanar to normal. In II three carboxylate groups are parallel to and two normal to the  $C_5H_5$  plane. Of the coplanar groups two are oriented such that the carbonyl O atoms are directed towards one another and chelate in two of the four coordination positions of Li ( $O-Li-O$  95.1(3)°). Coordination is completed by  $O(41)$  and  $H_2O$  ( $Li-O$  1.881(8)-1.914(7) Å). In III all the substituent carboxyl groups of the ligand are involved in coordination to potassiums whose environments consist of two pairs of chelating carboxylates from different ligands and an O atom from a further ligand. The sixth coordination site is occupied by methanol to form a distorted octahedral arrangement about K ( $K-O$  2.645(3)-2.884(5) Å). None of the substituent carboxyl groups in III are either parallel to or normal to the  $C_5H_5$  ring. In IV two of the carboxylate substituents are normal to the  $C_5H_5$  ring of the ligand while three are approximately coplanar. Two of the five coordination sites of Tl are occupied by carbonyl O atoms of adjacent carboxyls on one ligand while the other three sites are occupied by O atoms from three different ligands ( $Tl-O$  2.682(5)-3.244(5) Å). Five O atoms of each ligand are involved in coordination to four different Tl atoms. In each salt the geometry of the ligand is very similar to that in I.

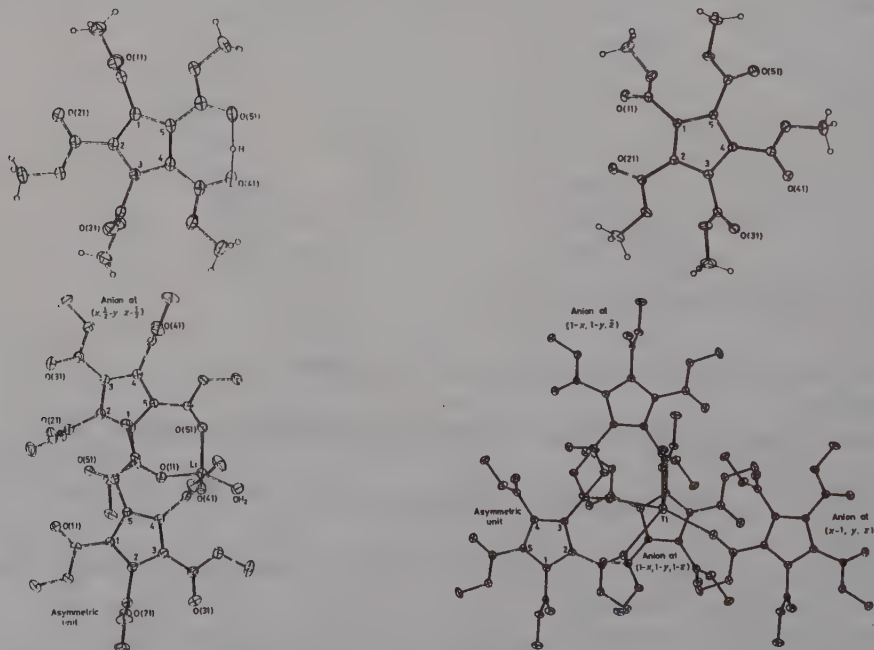
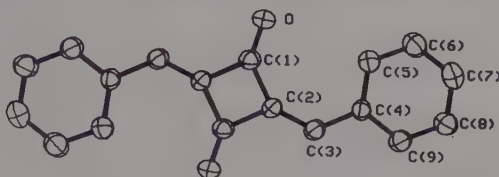


Fig. 1. The  $C_{15}H_{16}O_{10}$  molecule (top left), the molecular structure of  $C_{15}H_{15}LiO_{10} \cdot H_2O$  (bottom left), the anion in  $C_{15}H_{15}KO_{10} \cdot CH_4O$  (top right) and the Tl environment in  $C_{15}H_{15}O_{10}Tl$  (bottom right).

## (E)-2,4-BIS(BENZYLIDENE)CYCLOBUTANE-1,3-DIONE

 $C_{18}H_{12}O_2$ B.M. GATEHOUSE, 1982. Cryst. Struct. Comm., 11, 365-368.Monoclinic,  $P2_1/c$ ,  $a = 6.706$ ,  $b = 4.982$ ,  $c = 19.480$  Å,  $\beta = 95.08^\circ$ ,  $D_m = 1.34$ ,  $Z = 2$ . Cu radiation,  $R = 0.048$  for 972 reflexions.The molecule (Fig. 1) is centrosymmetric and nearly planar (torsion angle C2-C3-C4-C5  $-2.0(6)^\circ$ ). Bond lengths in the four-membered ring are C1-C2 1.491(5), C1-C2' 1.495(5) Å.Fig. 1. A view of  $C_{18}H_{12}O_2$ .

## r-1-CYANO-c-2-p-DIMETHYLAMINOPHENYL-1-PHENYLCYCLOPROPANE

 $C_{18}H_{18}N_2$  (I)

## r-1-CYANO-c-2-p-DIMETHYLAMINOPHENYL-1-p-NITROPHENYLCYCLOPROPANE

 $C_{18}H_{17}N_3O_2$  (II)

## r-1-CYANO-c-2-p-DIMETHYLAMINOPHENYL-t-3-METHYL-1-p-NITROPHENYLCYCLOPROPANE

 $C_{19}H_{19}N_3O_2$  (III)r-1-CYANO-c-2-(p-DIMETHYLAMINO- $\alpha$ -METHYLBENZYL)-t-3-METHYL-1-p-NITROPHENYLCYCLOPROPANE $C_{21}H_{23}N_3O_2$  (IV)

## r-1-CYANO-1-p-NITROPHENYL-c-2-PHENYLCYCLOPROPANE

 $C_{16}H_{12}N_2O_2$  (V)

## r-1-CYANO-t-3-METHYL-1-p-NITROPHENYL-c-2-PHENYLCYCLOPROPANE (VI)

 $C_{17}H_{14}N_2O_2$  (VI)B. TINANT, I. DE BLOCK, J.P. DECLERCQ, G. GERMAIN, M. VAN MEERSSCHE, G. LEROY and J. WEILER, 1982. Bull. Soc. Chim. Belg., 91, 629-639.I. Orthorhombic,  $P2_12_12_1$ ,  $a = 20.537$ ,  $b = 9.010$ ,  $c = 7.956$  Å,  $Z = 4$ . Mo radiation,  $R = 0.059$  for 578 reflexions.II. Triclinic,  $P\bar{1}$ ,  $a = 14.547$ ,  $b = 8.521$ ,  $c = 6.402$  Å,  $\alpha = 93.59$ ,  $\beta = 96.37$ ,  $\gamma = 88.30^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.058$  for 1780 reflexions.III. Triclinic,  $P\bar{1}$ ,  $a = 9.873$ ,  $b = 10.134$ ,  $c = 9.335$  Å,  $\alpha = 73.10$ ,  $\beta = 84.07$ ,  $\gamma = 98.68^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.068$  for 1742 reflexions.IV. Monoclinic,  $C2/c$ ,  $a = 23.837$ ,  $b = 12.389$ ,  $c = 14.374$  Å,  $\beta = 113.82^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.058$  for 1451 reflexions.V. Monoclinic,  $P2_1/c$ ,  $a = 7.152$ ,  $b = 17.814$ ,  $c = 10.717$  Å,  $\beta = 96.40^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.116$  for 1574 reflexions.VI. Monoclinic,  $P2_1/a$ ,  $a = 7.783$ ,  $b = 24.492$ ,  $c = 8.501$  Å,  $\beta = 115.59^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.070$  for 1844 reflexions.

The mean C-C bond lengths in the cyclopropane ring in I - VI are 1.501(7), 1.520(2), 1.556(5), 1.516(3), 1.522(6), 1.524(2) Å respectively. Substituent

induced bond length asymmetries observed in these cyclopropane derivatives are in good agreement with previous predictions. There is no evidence of conjugation transmission from an electron donor substituent to an acceptor one through the cyclopropane ring.

$N^{(1)}, N^{(2)}$ -BIS[2-METHYLTHIOTHIOCARBONYLCYCLOPENT-1-EN-1-YL]- $\omega, \omega'$ -DIETHYLENETRIAMINE  
 $C_{18}H_{29}N_3S_4$

P.B. SARKAR and S.P. SEN GUPTA, 1982. Cryst. Struct. Comm., 11, 433-438.

Triclinic,  $P\bar{1}$ ,  $a = 9.823$ ,  $b = 14.001$ ,  $c = 7.628$  Å,  $\alpha = 95.02^\circ$ ,  $\beta = 97.64^\circ$ ,  $\gamma = 87.72^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.062$  for 3329 reflexions.

In the molecule (Fig. 1), five-membered rings C(3)-C(7) and C(12)-C(16) are not individually planar; ring C(3)-C(7) is distorted towards an envelope conformation and ring C(12)-C(16) has a conformation in between half-chair and envelope. Torsion angles in the connecting chain are C(7)-N(1)-C(8)-C(9)  $179.6^\circ$  and C(9)-N(2)-C(10)-C(11)  $-176.0^\circ$ ; other torsion angles of the chain are all gauche. There are intra-molecular N-H...S hydrogen bonds (N...S 3.013 and 3.053 Å).

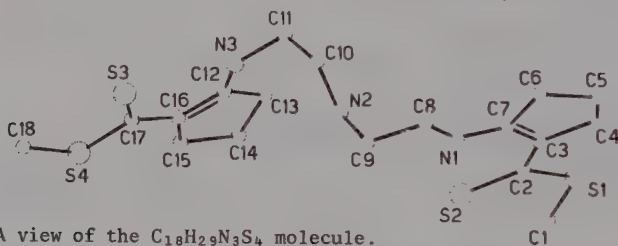


Fig. 1. A view of the  $C_{18}H_{29}N_3S_4$  molecule.

1,2-DI-*t*-BUTYL-3,4-DIISOPROPYLIDENE-1-CYCLOBUTENE  
 $C_{18}H_{30}$

H.J.B. SLOT, J. KROON, J.M. OOSTVEEN, H.J.T. BOS and P. VERMEER, 1982. Cryst. Struct. Comm., 11, 741-746.

Monoclinic,  $C2/c$ ,  $a = 14.922$ ,  $b = 9.412$ ,  $c = 11.394$  Å,  $\beta = 92.04^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.062$  for 1842 reflexions.

The four-membered ring is not planar (Fig. 1) with a mean deviation of 0.09 Å. A twofold crystallographic symmetry axis passes through the mid-points of the C1-C1A and C2-C2A bonds. A number of short C...C distances contribute to the puckering of this overcrowded molecule (C9...C9A 3.23, C5...C8 3.44, C7...C7A 3.50 Å).

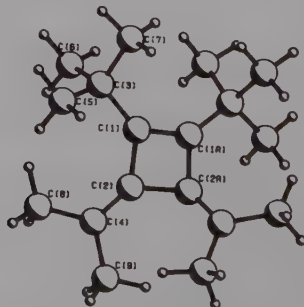


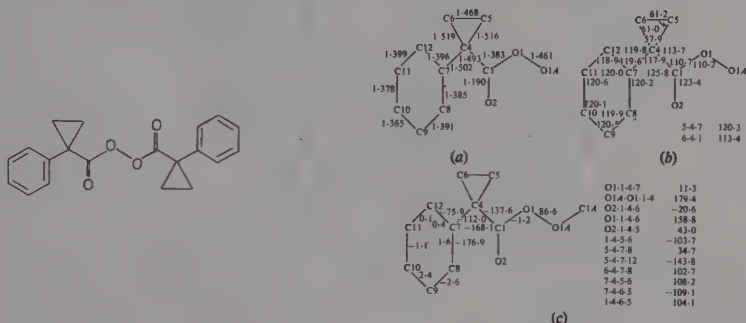
Fig. 1. The conformation of  $C_{18}H_{30}$ .

BIS[(1-PHENYL-1-CYCLOPROPYL)CARBONYL] PEROXIDE  
 $C_{20}H_{18}O_4$

D. BETHELL, D.J. CHADWICK, M.M. HARDING and G.Q. MALING, 1982. *Acta Cryst.*, **B38**, 1677-1679.

Monoclinic,  $C2/c$ ,  $a = 14.65$ ,  $b = 11.21$ ,  $c = 11.10$  Å,  $\beta = 106.6^\circ$ ,  $D_m = 1.25$ ,  $Z = 4$ .  
 Cu radiation,  $R = 0.060$  for 765 reflexions.

The molecule has  $\bar{1}$  crystallographic symmetry. The O(1)-O(1A) bond length (Fig. 1) is normal; the C(5)-C(6) bond is shortened compared to C(4)-C(5) and C(4)-C(6) indicating donation of electron density from cyclopropane to the attached  $\pi$  system. The carbonyl group adopts a bisected ( $\tau = 0^\circ$ ) conformation with O(2) lying over the cyclopropyl ring. The phenyl ring is oriented with an angle  $\tau = 69^\circ$  between the phenyl and cyclopropyl rings.



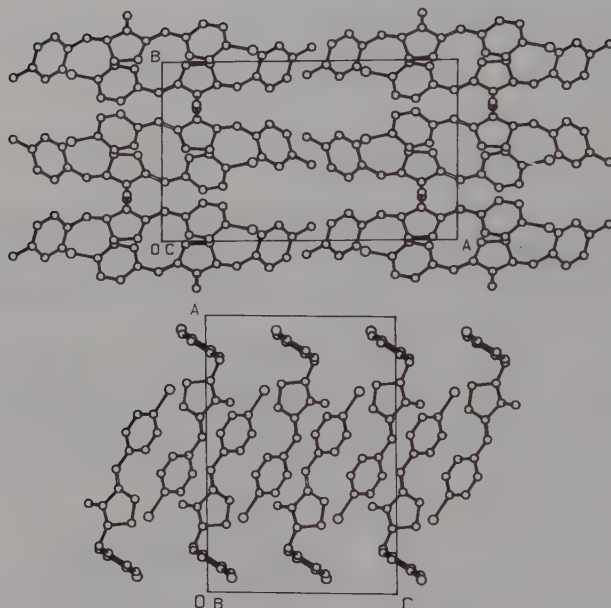


Fig. 1. Packing of the methyl- (top) and chloro- (lower) cyclopentanone isomers.

$N^{(\omega)}, N^{(\omega')}$ -BIS[2-METHYLTHIOTHIOCARBONYLCYCLOPENT-1-EN-1-YL]- $\omega, \omega'$ -TRIETHYLENETETRAMINE  
 $C_{20}H_{34}N_4S_4$

P.B. SARKAR and S.P. SEN GUPTA, 1982. Cryst. Struct. Comm., 11, 439-444.

Monoclinic,  $P2_1/c$ ,  $a = 8.186$ ,  $b = 18.454$ ,  $c = 16.263$  Å,  $\beta = 102.1^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.065$  for 2961 reflexions.

The two cyclopentene rings (Fig. 1) are not individually planar, but adopt envelope conformations. In the chain linking these rings, torsion angles are  $N(1)-C(8)-C(9)-N(2)$   $166.2^\circ$ ,  $C(8)-C(9)-N(2)-C(10)$   $177.5^\circ$ ,  $C(9)-N(2)-C(10)-C(11)$   $171.2^\circ$ ,  $C(11)-N(3)-C(12)-C(13)$   $178.7^\circ$ ,  $C(12)-C(13)-N(4)-C(14)$   $173.1^\circ$ ; other torsion angles are gauche. There are intramolecular  $N-H \cdots S$  hydrogen bonds ( $N(1) \cdots S(2)$  2.998,  $N(4) \cdots S(3)$  3.052 Å).

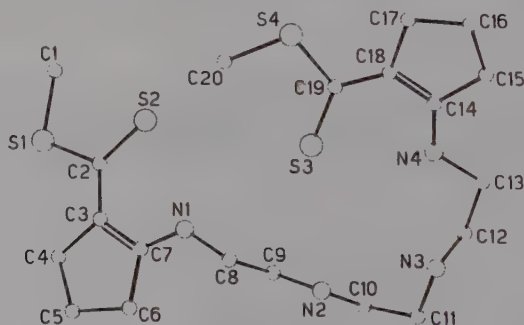
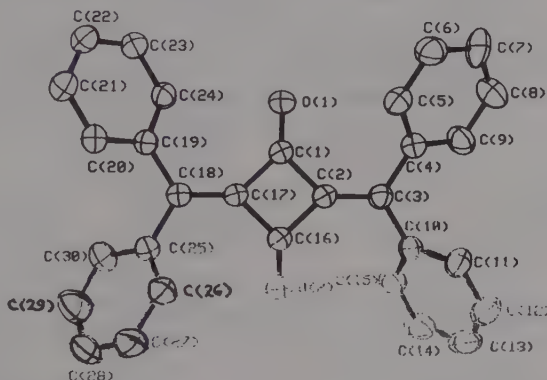


Fig. 1. A view of the  $C_{20}H_{34}N_4S_4$  molecule.

## 2,4-BIS (DIPHENYLMETHYLENE)CYCLOBUTANE-1,3-DIONE

 $C_{30}H_{20}O_2$ B.M. GATEHOUSE, 1982. Cryst. Struct. Comm., 11, 369-374.Monoclinic,  $P2_1/n$ ,  $a = 25.553$ ,  $b = 8.431$ ,  $c = 9.972$  Å,  $\beta = 95.01^\circ$ ,  $D_m = 1.26$ ,  $Z = 4$ .  
Cu radiation,  $R = 0.045$  for 3158 reflexions.

The molecule (Fig. 1) is almost symmetrical; there is a slight tetrahedral distortion of the central ring, with O(1) and O(2) being above the plane (0.083(3) and 0.079(3) Å respectively) through O(1), O(2), C(1), C(2), C(3), C(16), C(17), C(18), and C(13) and C(18) below it (-0.112(3) and -0.121(3) Å respectively). The phenyl groups are all rotated in the same sense with respect to the central ring which has mean C-C 1.500(4) Å and mean exocyclic C=C 1.363(4) Å.

Fig. 1. A view of  $C_{30}H_{20}O_2$ .

## BARIUM BIS[PENTAKIS(METHOXYCARBONYL)CYCLOPENTADIENIDE]

 $C_{30}H_{30}BaO_{20}$ 

M.I. BRUCE, J.K. WALTON, B.W. SKELTON and A.H. WHITE, 1982. J. Chem. Soc. Dalton, 2221-2226.

Triclinic,  $P\bar{1}$ ,  $a = 14.499$ ,  $b = 12.940$ ,  $c = 10.745$  Å,  $\alpha = 100.54$ ,  $\beta = 100.02$ ,  $\gamma = 114.23^\circ$ ,  $D_m = 1.62$ ,  $Z = 2$ . Mo radiation,  $R = 0.036$  for 8173 reflexions.

The Ba atom is eight-coordinate (Fig. 1) with an environment made up of three pairs of chelating carboxyl-carbonyl O atoms and two others (Ba-O 2.655(3)-2.932(3) Å). In the ligands two of the COOMe substituents on the  $C_5H_5$  ring are approximately perpendicular to the ring plane while the others are approximately parallel to this plane.



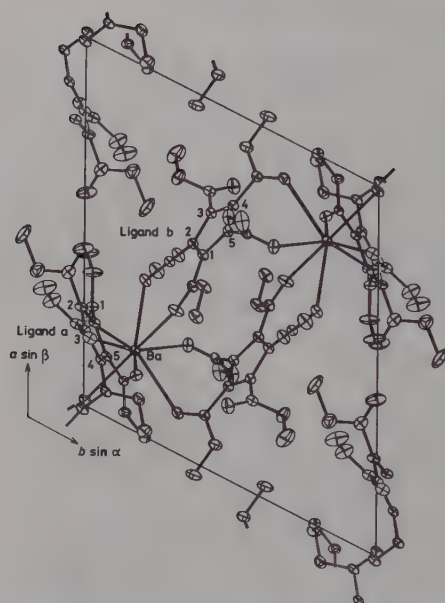


Fig. 1. The unit cell contents of  $C_{30}H_{30}BaO_{20}$  projected down  $c$ .

1-[2-BENZYL-1-PHENYLHYDRAZINOCARBONYL]-3-[2-BENZYL-1-PHENYLHYDRAZINOMETHYL]-1-BROMO-3-METHOXYCARBONYLCYCLOPROPANE  
 $C_{33}H_{29}BrN_4O_3$

B. FOUCHET, M. JOUCLA, J.C. MESSEGER and L. TOUPET, 1982. J. Chem. Soc. Chem. Commun., 858-859.

Triclinic,  $P\bar{1}$ ,  $a = 10.643$ ,  $b = 11.373$ ,  $c = 13.030$  Å,  $\alpha = 85.39$ ,  $\beta = 79.43$ ,  $\gamma = 70.70^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.032$  for 3920 reflexions.

The X-ray analysis established which ester group was substituted by the hydrazino group (Fig. 1). Selected bond distances and angles are: C(1)-C(2) 1.488(6), C(2)-C(3) 1.523(6), C(1)-C(3) 1.547(6), C(3)-C(4) 1.532(6) Å, C(2)-C(1)-C(3) 60.2(3), C(1)-C(2)-C(3) 61.8(3) and C(1)-C(3)-C(2) 58.0(3)°.

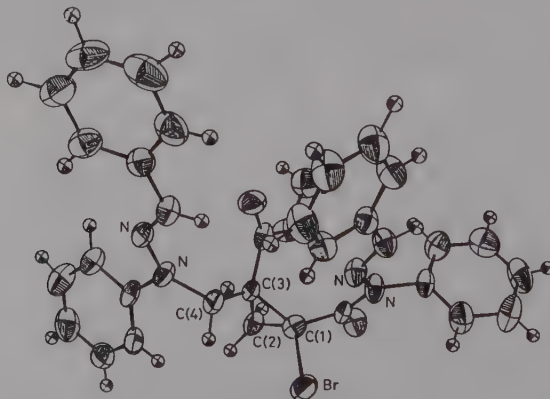


Fig. 1.  $C_{33}H_{29}BrN_4O_3$ : the molecular structure.

3,4-trans-5-TRIBROMO-cis-6-HYDROXY-2,6-DIMETHYL-R-2,5-DINITROCYCLOHEX-3-ENONE  
 $C_8H_7Br_3N_2O_6$  (I)

3,4-cis-5-TRIBROMO-trans-6-HYDROXY-2,6-DIMETHYL-R-2,5-DINITROCYCLOHEX-3-ENONE  
 $C_8H_7Br_3N_2O_6$  (II)

A.M. CHITTENDEN, M.P. HARTSHORN, K.E. RICHARDS, W.T. ROBINSON, K.H. SUTTON, R.S. THOMPSON and J. VAUGHAN, 1982. *Aust. J. Chem.*, **35**, 2229-2236.

I. Monoclinic,  $P2_1/c$ ,  $a = 13.187$ ,  $b = 9.113$ ,  $c = 11.043$  Å,  $\beta = 95.91^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.041$  for 825 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 12.474$ ,  $b = 6.266$ ,  $c = 16.821$  Å,  $\beta = 91.00^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.050$  for 736 reflexions.

In both structures (Fig. 1) bond lengths and angles have normal values. The O(1)...O(6) distance (2.65(3) in I and II) is consistent with an intramolecular hydrogen bond although the H atoms were not located in the analysis. The conformation of each ring is a twist-boat (C(4)C(3)-C(2)C(1)  $-1(2)^\circ$  (I),  $-10(2)^\circ$  (II); C(3)C(4)-C(5)C(6)  $23(2)^\circ$  (I),  $23(2)^\circ$  (II)).

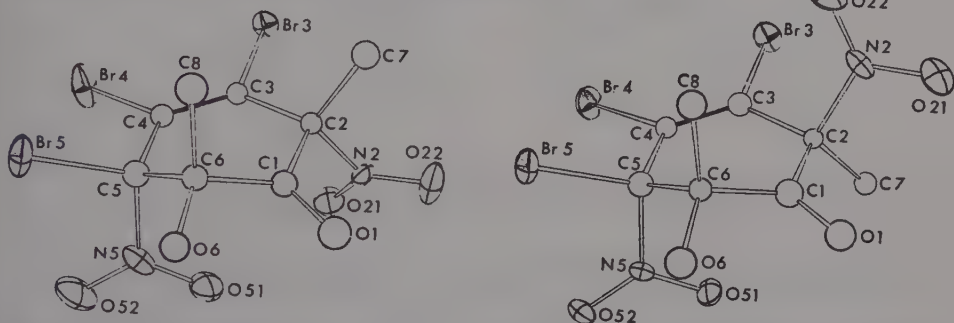
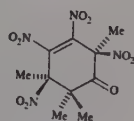


Fig. 1. Perspective views of the trans-cis isomer of  $C_8H_7Br_3N_2O_6$  (I) (left) and the cis-trans isomer  $C_8H_7Br_3N_2O_6$  (II) (right).

cis-2,5,6,6-TETRAMETHYL-2,3,4,5-TETRANITROCYCLOHEX-3-ENONE  
 $C_{10}H_{12}N_4O_9$



M.J. GRAY, M.P. HARTSHORN, K.E. RICHARDS, W.T. ROBINSON, K.H. SUTTON, R.S. THOMPSON and J. VAUGHAN, 1982. *Aust. J. Chem.*, **35**, 1237-1243.

Monoclinic,  $P2_1/c$ ,  $a = 9.081$ ,  $b = 12.994$ ,  $c = 12.897$  Å,  $\beta = 112.98^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.091$  for 341 reflexions.

The structure was confirmed as that shown above.

cis-1,2,3,4,5,6-HEXAMETHYLCYCLOHEXANE  
 $C_{12}H_{24}$

H. VAN KONINGSVELD, J.M.A. BAAS, B. VAN DE GRAAF and M.A. HOEFNAGEL, 1982. *Cryst. Struct. Comm.*, **11**, 1065-1071.

Monoclinic,  $P2_1/n$ ,  $a = 6.416$ ,  $b = 13.612$ ,  $c = 13.080$  Å,  $\beta = 103.59^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.039$  for 2323 reflexions (at  $-160^\circ\text{C}$ ).

The molecule has chair form with methyl groups alternately axial and equatorial. In the cyclohexane ring C-C distances are 1.540-1.548(2) Å, axial C-C 1.534-1.536(2). equatorial C-C 1.526-1.531(3) Å.

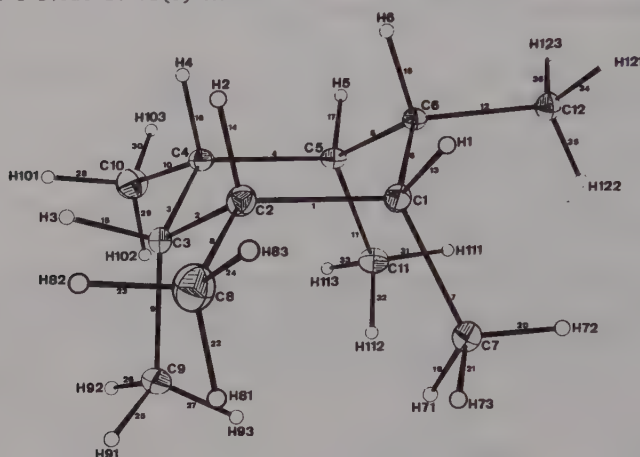


Fig. 1. A view of  $C_{12}H_{24}$ .

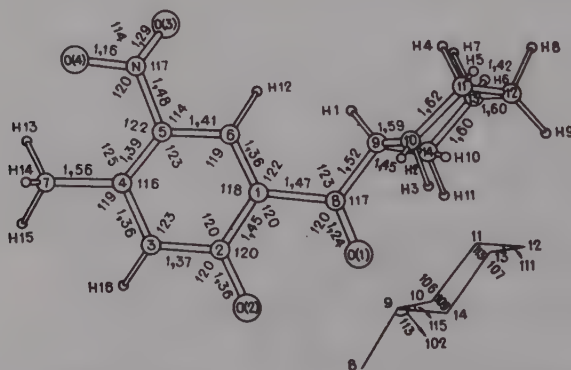
2-HYDROXY-4-METHYL-5-NITROPHENYL-CYCLOHEXYLKETONE

$C_{14}H_{17}NO_4$

T. ATABAEV, Yu.V. GATILOV, N.V. PODBEREZSKAYA, S.V. BORISOV and A. ASHIROV, 1982. Zh. Strukt. Khim., 23-4, 166-168 [J. Struct. Chem., 23, 638-639].

Monoclinic,  $P2_1$ ,  $a = 13.243$ ,  $b = 6.900$ ,  $c = 7.577$  Å,  $\beta = 107.37^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.129$  for 710 reflexions.

The geometry of the molecule is shown in Fig. 1 ( $\sigma = 0.01$ - $0.04$  Å). With the exception of C(7), O(3), and the cyclohexyl carbon atoms; the molecule is planar within 0.06 Å.



3,3,6,6-TETRAMETHOXY-1,2,4,5-TETRAMETHYL-1,4-CYCLOHEXADIENE  
 $C_{14}H_{24}O_4$

L. NORSKOV-LAURITSEN, S. LARSEN, M.G. ETTLINGER and J.W. JAROSZEWSKI, 1982. Acta Cryst., B38, 3107-3110.

Monoclinic, C2/c,  $a = 14.043$ ,  $b = 11.506$ ,  $c = 8.851$  Å,  $\beta = 104.94^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.046$  for 2231 reflexions (at 90 K).

The molecule (Fig. 1) lies about an inversion centre and the conformation of the dimethyl acetal groups is anti,anti. The O-C-O angle is  $99^\circ$ , C(1)=C(2) 1.343(1), C-O 1.423-1.435(1) Å.

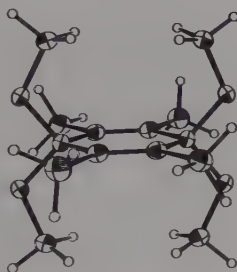


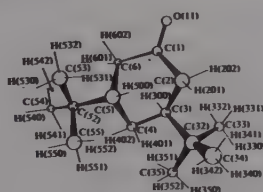
Fig. 1. A view of  $C_{14}H_{24}O_4$ .

trans-3,5-DI-t-BUTYLCYCLOHEXANONE  
 $C_{14}H_{26}O$

S. TOURÉ, J. LAPASSET, B. BOYER and G. LAMATY, 1982. Acta Cryst., B38, 1383-1385.

Monoclinic, C2/c,  $a = 25.136$ ,  $b = 6.284$ ,  $c = 18.864$  Å,  $\beta = 107.98^\circ$ ,  $Z = 8$ .  $R = 0.075$  for 2056 reflexions.

In the molecule (Fig. 1) the ring has a twist conformation with a non-crystallographic  $C_2$  symmetry axis through C(1) and C(4). Bond lengths and angles are generally as expected. Intermolecular interactions correspond to van der Waals contacts.



C(1)-C(2)	1.495 (5)	C(32)-C(33)	1.531 (6)
C(2)-C(3)	1.537 (5)	C(32)-C(34)	1.539 (6)
C(3)-C(4)	1.542 (5)	C(32)-C(35)	1.538 (5)
C(4)-C(5)	1.545 (5)	C(5)-C(52)	1.546 (5)
C(5)-C(6)	1.534 (5)	C(52)-C(53)	1.545 (7)
C(6)-C(1)	1.506 (6)	C(52)-C(54)	1.529 (6)
C(1)-O(11)	1.223 (4)	C(52)-C(55)	1.529 (6)
C(3)-C(32)	1.552 (5)		

Fig. 1.  $C_{14}H_{26}O$ : a perspective view of the molecule and bond lengths.

5,5-DIMETHYLCYCLOHEXANE-1,2,3-TRIONE 2-(4-METHYLPHENYLHYDRAZONE)  
 $C_{15}H_{18}N_2O_2$  (I)

5,5-DIMETHYLCYCLOHEXANE-1,2,3-TRIONE 2-(4-NITROPHENYLHYDRAZONE)  
 $C_{14}H_{15}N_3O_4$  (II)

M.G.B. DREW, B. VICKERY and G.R. WILLEY, 1982. J. Chem. Soc. Perkin II, 1297-1303.

I. Monoclinic,  $P2_1/n$ ,  $a = 14.053$ ,  $b = 5.84$ ,  $c = 16.789$  Å,  $\beta = 98.9^\circ$ ,  $D_m = 1.43$ ,  $Z = 4$ . Mo radiation,  $R = 0.087$  for 853 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 7.20$ ,  $b = 13.45$ ,  $c = 7.45$  Å,  $\alpha = 97.97$ ,  $\beta = 94.24$ ,  $\gamma = 98.29^\circ$ ,  $D_m = 1.48$ ,  $Z = 2$ . Mo radiation,  $R = 0.078$  for 1638 reflexions.

Both structures (Fig. 1) contain an intramolecular hydrogen bond between O(11) and H(7), with N(7)...O(11) 2.63 Å in both I and II. The N(8)-C(9)-C(10) bond angle is greater than N(8)-C(9)-C(20) (respectively 123.3, 114.7 in I, and 125.1, 114.3° in II) as a result of the requirements of the O(11)...H(7) hydrogen bond. The phenylhydrazone fragment is essentially planar in both structures with the nitro group in II twisted 8.3° out of the plane of the aromatic ring. The bond lengths in the hydrazone link are, for I and II respectively, C(9)-N(8) 1.336, 1.308, N(8)-N(7) 1.307, 1.314 and N(7)-C(4) 1.429, 1.405 Å. The C(20)-O(21) and C(10)-O(11) bond lengths (Fig. 1) both indicate keto bonds and C(20)-C(9) and C(9)-C(10) are also similar, indicating an absence of enolisation in the structures (unlike dimedone itself). The envelope conformation of dimedone has been retained with slightly increased puckering of the cyclohexane ring at C(13). Deviations from the C(10), C(20), C(12), C(22) mean plane are C(9) 0.01, 0.02 and C(13) 0.68, 0.68 Å in I and II respectively. There are no short intermolecular contacts in the crystals and the different space groups and packing in I and II do not appear to influence the molecular conformations.

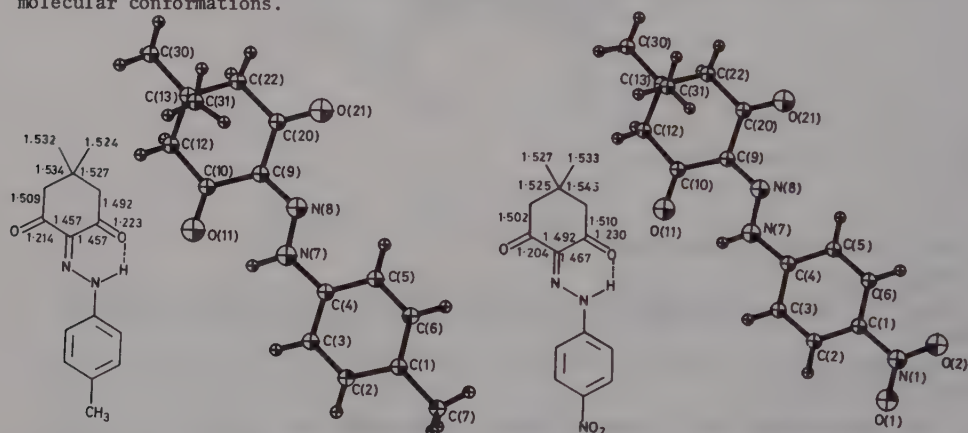


Fig. 1. Molecular skeletons and structures:  $C_{15}H_{18}N_2O_2$  (left) and  $C_{15}H_{15}N_3O_4$  (right).

## 1,4-trans-CYCLOHEXYLENE DIMETHYL DISUCCINATE



B. RÉMILLARD and F. BRISSE, 1982. *Acta Cryst.*, B38, 1220-1224.

Triclinic,  $P\bar{1}$ ,  $a = 6.169$ ,  $b = 7.276$ ,  $c = 10.360$  Å,  $\alpha = 89.32$ ,  $\beta = 84.30$ ,  $\gamma = 73.84^\circ$ ,  $D_m = 1.28$ ,  $Z = 1$ . Cu radiation,  $R = 0.046$  for 1332 reflexions.

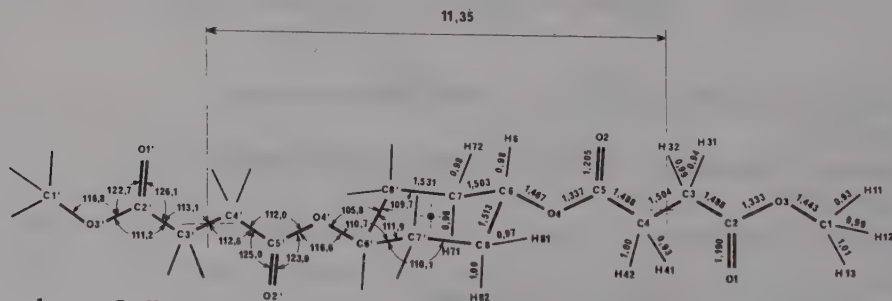


Fig. 1.  $C_{16}H_{24}O_8$ : bond distances and angles in the molecule.



The molecule (Fig. 1) lies on an inversion centre and the cyclohexane ring is in a chair conformation. In the crystal the molecules are all parallel to each other. The fibre repeat of the parent polyester would be close to 11.35 Å.

r-1,c-2,t-3,c-4,t-5,c-6-HEXAMETHOXYCARBONYLCYCLOHEXANE  
 $C_{18}H_{14}O_{12}$  (I)

r-1-ETHOXYCARBONYL,c-2,t-3,c-4,t-5,c-6-PENTAMETHOXYCARBONYLCYCLOHEXANE  
 $C_{19}H_{26}O_{12}$  (II)

S. BRUCKNER, L. MALPEZZI and M. GRASSI, 1982. Cryst. Struct. Comm., **11**, 1043-1048.

I. Orthorhombic, Pbc<sub>a</sub>,  $a = 15.236$ ,  $b = 14.935$ ,  $c = 18.986$  Å,  $Z = 8$ . Mo radiation,  $R = 0.049$  for 1751 reflexions.

II. Monoclinic, P2<sub>1</sub>/c,  $a = 9.278$ ,  $b = 22.082$ ,  $c = 11.564$  Å,  $\beta = 112.70^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.056$  for 2014 reflexions.

Substitution of a methyl group by an ethyl group does not involve significant differences in intramolecular interactions. Views of the molecules are in Fig. 1 along with mean values for the methoxycarbonyl moieties.

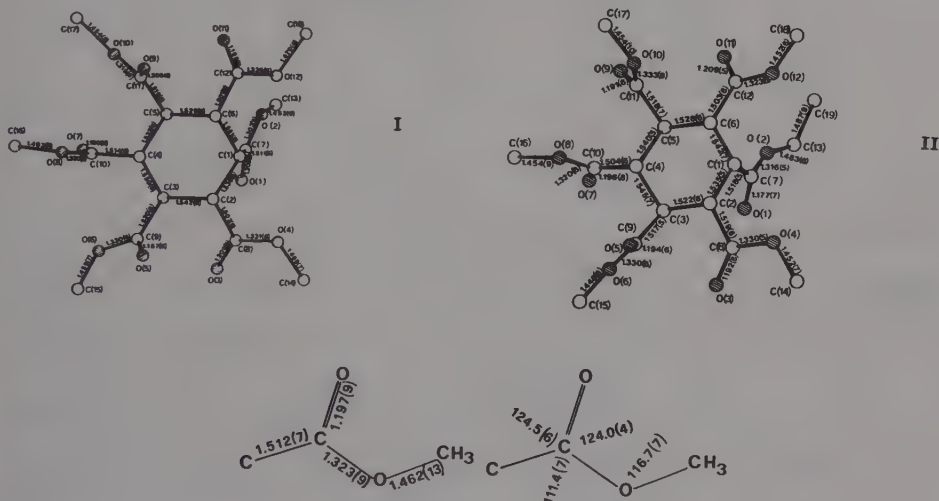


Fig. 1. Molecular geometry details for  $C_{18}H_{14}O_{12}$  and  $C_{19}H_{26}O_{12}$ , and mean methoxycarbonyl dimensions.

1-(1'-HYDROXY-1'-METHYL-2'-BENZOYLOXY)-ETHYL-1-HYDROXY-2,2,6-TRIMETHYLCYCLOHEXANE  
 $C_{19}H_{28}O_4$

B. NORBERG, F. DURANT, A. MICHEL and G. EVRARD, 1982. Cryst. Struct. Comm., **11**, 659-664.

Monoclinic, P2<sub>1</sub>/c,  $a = 18.967$ ,  $b = 8.183$ ,  $c = 11.462$  Å,  $\beta = 91.76^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.031$  for 1328 reflexions.

The molecular structure (Fig. 1) is characterized by an intramolecular O-H...O





In the crystal structure (Fig. 1) the cyclohexane moiety of the molecule is in the chair conformation about an inversion centre. The two bonds adjacent to the O atom are significantly shorter than the middle cyclohexane bonds. However, the two trans C-O bonds are not in the cyclohexane mirror plane, the two C-C-O angles having values of 106.1 and 110.4° respectively.

trans-4-t-BUTYLCYCLOHEXYL (2R)-3,3,3-TRIFLUORO-2-METHOXY-2-PHENYLPROPIONATE  
 $C_{20}H_{27}F_3O_3$

H.M. DOESBURG, G.H. PETIT and E.M. MERCKX, 1982. Acta Cryst., B38, 1181-1185.

Monoclinic,  $P2_1$ ,  $a = 6.993$ ,  $b = 11.288$ ,  $c = 12.303$  Å,  $\beta = 95.51^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.047$  for 2003 reflexions.

In the molecule (Fig. 1) the orientation of the trans-4-t-butylcyclohexyl residue with respect to the trifluoromethoxyphenylpropionic acid ester is different from that in other equatorial esters. The other geometrical parameters are unexceptional. The packing of the molecules in the crystal is determined by van der Waals forces.

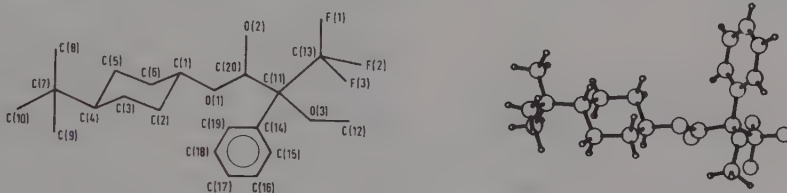


Fig. 1.  $C_{20}H_{27}F_3O_3$ : the molecular skeleton and a perspective view.

1,1,3,3-BIS(1',1',5',5'-TETRAMETHYLPENTAMETHYLENE) ALLENE  
 $C_{21}H_{36}$

$(C_{10}H_{18})_2C$

H. IRNGARTINGER, E. KURDA, H. RODEWALD, A. BERNOT, R. BOLZE and K. SCHÜLTER, 1982. Chem. Ber., 115, 967-972.

Monoclinic,  $P2_1/c$ ,  $a = 13.183$ ,  $b = 10.542$ ,  $c = 13.999$  Å,  $\beta = 91.97^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.055$  for 1685 reflexions.

The six-membered rings (Fig. 1) adopt chair conformations; the dihedral angle between the two mean ring planes is  $96.1^\circ$ .



## 1,4-trans-CYCLOHEXANEDIYLDIMETHYLENE DIBENZOATE

 $C_{22}H_{24}O_4$  (I) $(C_6H_5COOCH_2)_2C_6H_{10}$ 

## 1,4-trans-CYCLOHEXANEDIYLDIMETHYLENE 3,3'-BIS(METHOXYCARBONYL)DIPROPIONATE

 $C_{18}H_{28}O_8$  (II) $(CH_3OCOCH_2CH_2COOCH_2)_2C_6H_{10}$ 

F. BRISSE and B. RÉMILLARD, 1982. Acta Cryst., B38, 825-830.

I. Monoclinic,  $P2_1/c$ ,  $a = 9.399$ ,  $b = 11.192$ ,  $c = 9.006$  Å,  $\beta = 90.99^\circ$ ,  $D_m = 1.23$ ,  $Z = 2$ . Mo radiation,  $R = 0.039$  for 438 reflexions.II. Monoclinic,  $P2_1/c$ ,  $a = 11.490$ ,  $b = 9.322$ ,  $c = 9.151$  Å,  $\beta = 93.56^\circ$ ,  $D_m = 1.27$ ,  $Z = 2$ . Mo radiation,  $R = 0.037$  for 773 reflexions.

Bond distances and angles in I and II are given in Fig. 1. The parent polyesters are: poly(1,4-trans-cyclohexanediylldimethylene terephthalate) and poly(1,4-trans-cyclohexanediylldimethylene succinate). Their fibre repeats (assuming the model compound and the corresponding polymer have the same conformation) have the values of 14.33 and 13.45 Å respectively.

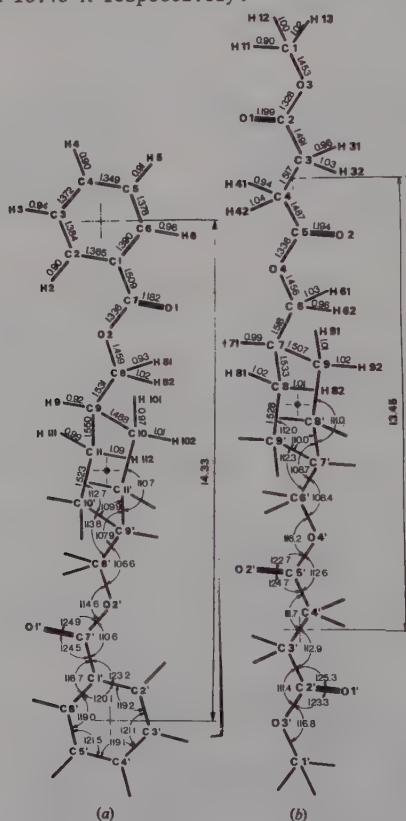


Fig. 1. Bond distances and angles in  $C_{22}H_{24}O_4$  (I) (left) and  $C_{18}H_{28}O_8$  (II) (right).

## 1-DIPHENYLMETHYLENE-4-TRIPHENYLMETHYL-2,5-CYCLOHEXADIENE

 $C_{38}H_{30}$ 

N.S. BLOM, G. ROELOFSEN and J.A. KANTERS, 1982. Cryst. Struct. Comm., 11, 297-304.

Triclinic,  $P\bar{1}$ ,  $a = 13.939$ ,  $b = 14.326$ ,  $c = 17.026$  Å,  $\alpha = 68.626$ ,  $\beta = 65.438$ ,  $\gamma = 66.907^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.061$  for 2567 reflexions.

There are two independent molecules (Fig. 1) and both show large C(4)-C(20) distances of 1.60 and 1.61 Å respectively. A three-atom fit of the two independent molecules shows an almost perfect resemblance of the molecules except for phenyl ring B. Apart from minor differences, the geometry of the triphenylmethyl tetrahedrons in the independent molecules are similar. The same holds for the torsion angles about C(4)-C(20), which have values close to that of the ideal staggered conformation. The cyclohexadiene rings are puckered with average deviations from the best plane of 0.05 and 0.06 Å respectively. The exocyclic methylene C is distorted from this plane by 0.2 and 0.3 Å respectively towards the triphenylmethyl group.

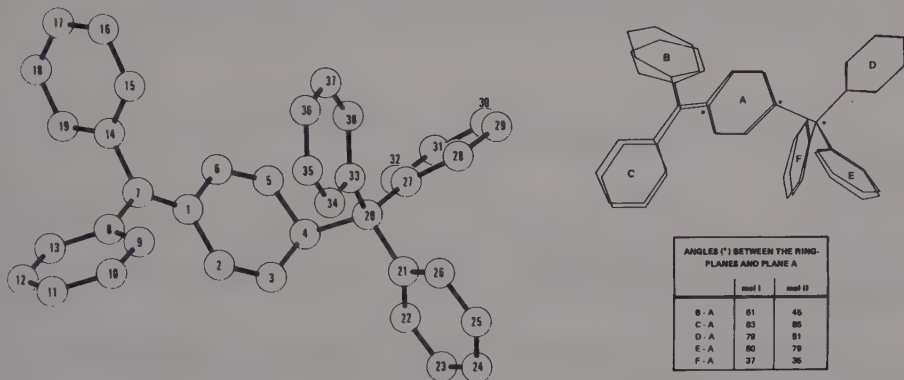


Fig. 1. One of the  $C_{38}H_{30}$  molecules and a three-atom best fit of the two molecules.

# OCTAFLUOROCYCLOOCTATETRAENE

$C_8F_8$

B.B. LAIRD and R.E. DAVIS, 1982. *Acta Cryst.*, B38, 678-680.

Monoclinic,  $P2_1/c$ ,  $a = 9.96$ ,  $b = 7.04$ ,  $c = 12.66$  Å,  $\beta = 111.6^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.044$  for 1337 reflexions at 238 K.

The  $C_8$  ring has a slightly more flattened, tub configuration than in  $C_8H_8$ . All F atoms tilt slightly outwards by about  $1.9^\circ$  from the tub defined by the carbon ring. The molecular geometry is shown in Fig. 1.

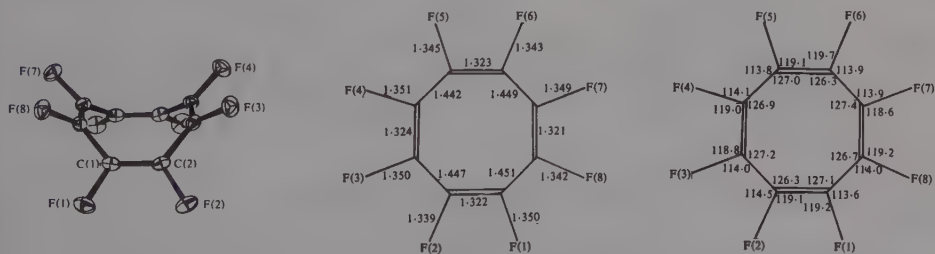
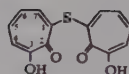


Fig. 1. Bond distances (e.s.d.'s 0.002-0.004 Å) and angles (e.s.d.'s  $0.2^\circ$ ) in octafluorocyclooctatetraene.

## BIS(3-HYDROXY-2-OXOCYCLOHEPTA-3,5,7-TRIENYL)SULPHIDE

 $C_{14}H_{10}O_4S$ 

K. GORLER, W. MOLLS, H.U. SIEHL, J. STRAHLE and C. WESTPHAL, 1982. Justus Liebigs Ann. Chem., 1006-1011.

Monoclinic,  $P2_1$ ,  $a = 7.612$ ,  $b = 13.265$ ,  $c = 11.812$  Å,  $\beta = 90.28^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.094$  for 1606 reflexions.

The two seven-membered rings are planar within experimental error and are twisted with respect to one another (dihedral angles between the ring planes are  $77.74$  and  $-73.57^\circ$  in the two independent molecules). The mean S-C distance is  $1.78(1)$  Å. The ring planarity and bond lengths indicate that the tautomer shown above predominates.

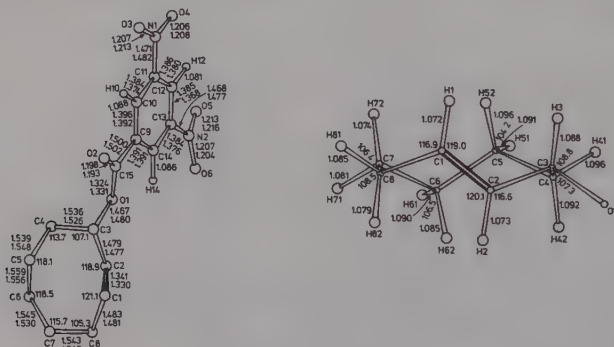
## trans-2-CYCLOOCTEN-1-YL 3,5-DINITROBENZOATE

 $C_{15}H_{16}N_2O_6$ 

O. ERMER and S.A. MASON, 1982. Acta Cryst., B38, 2200-2206.

Triclinic,  $P\bar{1}$ ,  $a = 10.963$ ,  $b = 5.656$ ,  $c = 14.873$  Å,  $\alpha = 104.91$ ,  $\beta = 122.75$ ,  $\gamma = 76.18^\circ$ ,  $D_m = 1.41$ ,  $Z = 2$ . Mo radiation,  $R = 0.052$  for 1623 reflexions; neutron diffraction,  $R = 0.030$  for 1824 reflexions.

In the molecule (Fig. 1) the trans-cyclooctene ring closely approximates  $C_2$  symmetry. The non-planar deformation of the trans double bond is composed of  $19.6(3)^\circ$  twisting and  $22.3(3)^\circ$  ( $C_2$  average) out-of-plane bending; the C-C=C torsion angle is  $138.1(1)^\circ$  (neutron results).



In the molecule (Fig. 1) the cyclooctatetraenyl group has typical dimensions for a monosubstituted derivative. Bond lengths are S-S 2.044, mean C-S 1.784, and, in the cyclooctatetraenyl group, mean C-C 1.482 and mean C=C 1.328 Å. The normals to the two benzene-ring planes intersect at 82°.

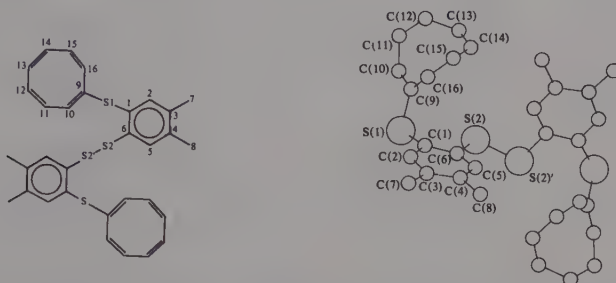


Fig. 1. The  $C_{32}H_{30}S_4$  molecule viewed normal to the plane S(1)C(9)C(14).

# 1,8-DIMETHYL[14]ANNULENE

$C_{16}H_{18}$

E. VOGEL, H.-W. ENGELS, W. HUBER, J. LEX and K. MÜLLEN, 1982. J. Am. Chem. Soc., **104**, 3729-3731.

Monoclinic, C2/c,  $a = 19.010$ ,  $b = 4.178$ ,  $c = 15.926$  Å,  $\beta = 98.70^\circ$ ,  $D_m = 1.10$ ,  $Z = 4$ . Mo radiation,  $R = 0.069$  for 497 reflexions.

The non-planar centrosymmetric molecule is shown in Fig. 1. Bond lengths are typical of benzenoid aromatic bonds although intraannular torsion angles are as large as  $20^\circ$ .

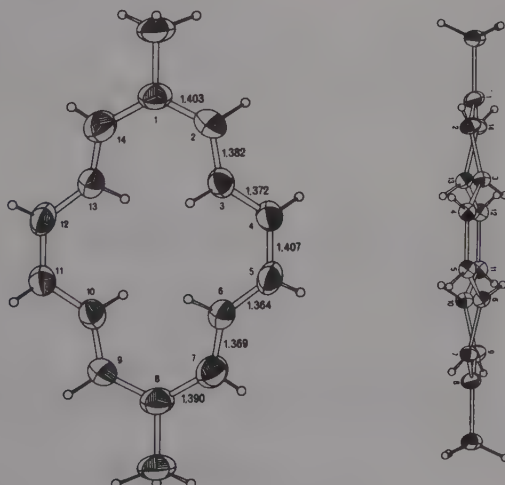


Fig. 1. Two views of 1,8-dimethyl[14]annulene.



## cis-CIVETONE

 $C_{17}H_{30}O$  (I)cis-CIVETONE 2,4-DINITROPHENYLHYDRAZONE ( $\alpha$  and  $\beta$  Forms) $C_{23}H_{34}N_4O_4$  (II, III)G. BERNARDINELLI and R. GERDIL, 1982. *Helv. Chim. Acta*, **65**, 558-572.

I. Tetragonal,  $I4_1$ ,  $a = 9.95$ ,  $c = 32.79 \text{ \AA}$ ,  $D_m = 0.91$ ,  $Z = 8$ . Mo radiation, 233 reflexions (at 153 K).

II. Triclinic,  $P\bar{1}$ ,  $a = 6.279$ ,  $b = 12.605$ ,  $c = 15.253 \text{ \AA}$ ,  $\alpha = 105.49$ ,  $\beta = 100.31$ ,  $\gamma = 91.23^\circ$ ,  $D_m = 1.19$ ,  $Z = 2$ . Mo radiation,  $R = 0.11$  for 1421 reflexions (at 173 K).

III. Triclinic,  $P\bar{1}$ ,  $a = 7.950$ ,  $b = 8.405$ ,  $c = 18.233 \text{ \AA}$ ,  $\alpha = 100.28$ ,  $\beta = 92.29$ ,  $\gamma = 94.18^\circ$ ,  $D_m = 1.22$ ,  $Z = 2$ . Mo radiation,  $R = 0.11$  for 1656 reflexions.

Civetone was examined in its plastic phase. The molecules appear as ring shaped diffuse electron-density distributions. Derivatives II and III were well resolved (Fig. 1). The polymorphs have different macrocycle ring conformations,  $\alpha$  [2513\*4\*4],  $\beta$  [25134\*4\*].

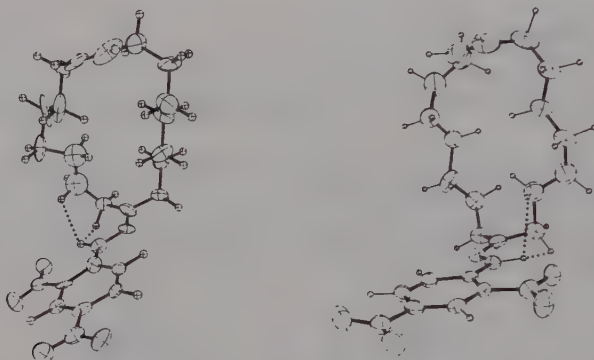


Fig. 1. Views of the  $\alpha$ - and  $\beta$ - forms of  $C_{23}H_{34}N_4O_4$ .

## 1,10-CYCLOOCTADECANEDIONE

 $C_{18}H_{32}O_2$ 

N.L. ALLINGER, B.J. GORDEN, M.G. NEWTON, L. NORSKOV-LAURITSEN and S. PROFETA, JR., 1982. *Tetrahedron*, **38**, 2905-2909.

Orthorhombic,  $Cmca$ ,  $a = 17.663$ ,  $b = 9.765$ ,  $c = 10.233 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.058$  for 642 reflexions.

The molecule (Fig. 1) has crystallographic  $C_{2h}$  symmetry. The carbon atoms are arranged in a diamondoid lattice. The dipole moment for the solid state conformation is zero while in solution it is 2.78D.

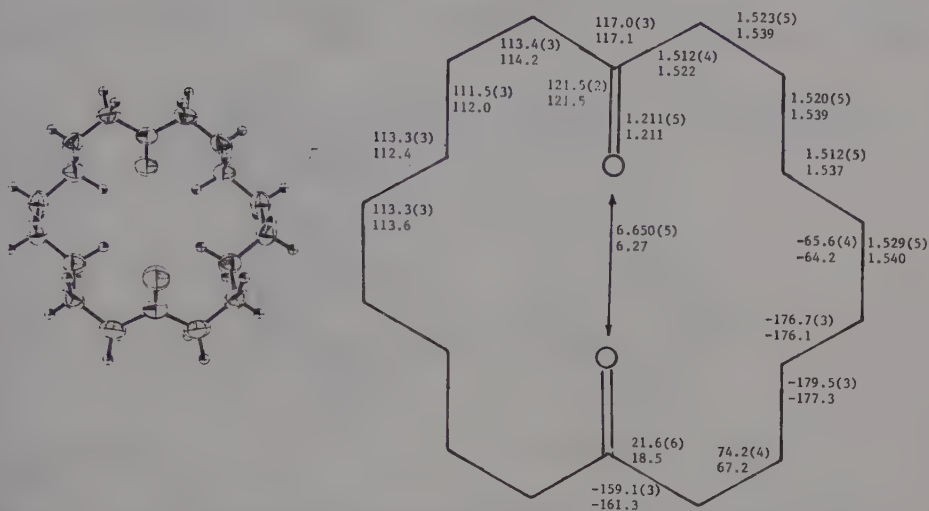


Fig. 1. The  $C_{18}H_{32}O_2$  molecule and dimensions (the values without error estimates are from molecular mechanics calculations).

### 3-METHYLCYCLOPENTADECANONE 2,4-DINITROPHENYLHYDRAZONE

$C_{22}H_{34}N_4O_4$

G. BERNARDINELLI and R. GERDIL, 1982. *Helv. Chim. Acta*, **65**, 1310-1317.

Triclinic,  $P\bar{1}$ ,  $a = 8.015$ ,  $b = 8.235$ ,  $c = 17.409$  Å,  $\alpha = 107.69$ ,  $\beta = 93.86$ ,  $\gamma = 96.03^\circ$ ,  $D_m = 1.22$ ,  $Z = 2$ . Mo radiation,  $R = 0.155$ .

The macrocycle (Fig. 1) shows static disorder which was partially resolved by analysis of the anisotropic vibration parameters. Eight possible conformers with reasonable geometries emerged from the refinements and were used as starting models in force-field calculations.

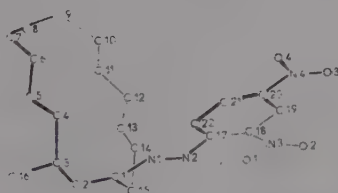


Fig. 1. A view of  $C_{22}H_{34}N_4O_4$ .

### trans-CIVETONE 2,4-DINITROPHENYLHYDRAZONE

$C_{23}H_{34}N_4O_4$

G. BERNARDINELLI and R. GERDIL, 1982. *Helv. Chim. Acta*, **65**, 730-738.

Monoclinic,  $P2_1/c$ ,  $a = 8.364$ ,  $b = 7.971$ ,  $c = 36.025$  Å,  $\beta = 91.44^\circ$ ,  $D_m = 1.204$ ,  $Z = 4$ . Mo radiation,  $R = 0.09$  for 2286 reflexions.

The civetone molecule adopts the conformation described as  $[23^*434^*44^*]$  and

shown in Fig. 1. Its mean plane is approximately perpendicular to that of the aromatic substituent.

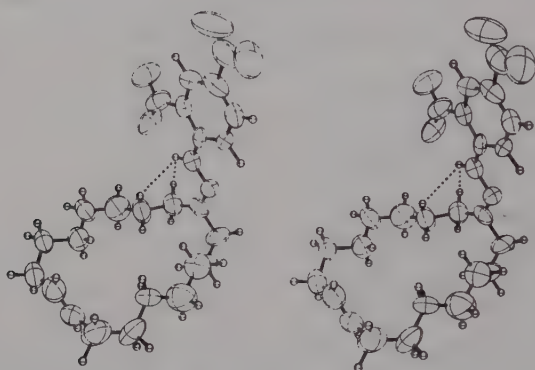


Fig. 1. A stereoview of  $C_{23}H_{34}N_4O_4$ .

1,2,9,10,17,18-HEXADEHYDRO[2.2.2]PARACYCLOPHANE  
 $C_{24}H_{18}$

K. TRUEBLOOD, K. MIRSKY, E. MAVERICK, C. KNOBLER and L. GROSSENBACHER, 1982. Acta Cryst., B38, 2428-2435.

Monoclinic,  $P2_1/n$ ,  $a = 16.914$ ,  $b = 10.066$ ,  $c = 10.292$  Å,  $\beta = 105.90^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.086$  for 2325 reflexions (at 113 K).

Monoclinic,  $C2/c$ ,  $a = 17.111$ ,  $b = 10.132$ ,  $c = 10.581$  Å,  $\beta = 106.95^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.103$  for 886 reflexions (at 298 K).

The structure analyses and potential energy calculations are consistent with an ordered structure at 113 K in which individual rings are librating and the whole molecule librates as well (Fig. 1). The aromatic rings are bent to a small extent into a boat shape but bond lengths and angles are quite normal. At 298 K there is increased molecular motion and the calculations indicate that the observed structure is a dynamic average of two equivalent molecules related by a twofold axis.

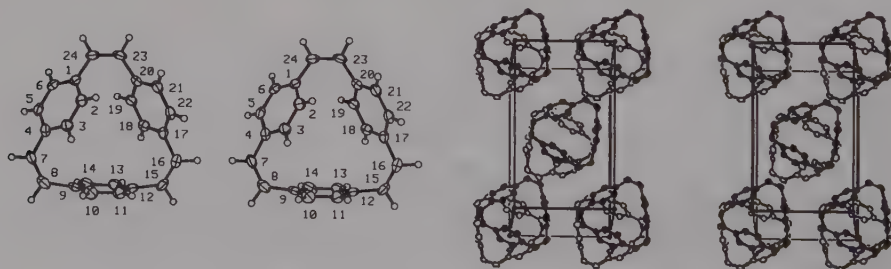


Fig. 1.  $C_{24}H_{18}$ : stereoviews of the molecule and of the crystal structure at 113 K.

CYCLOHEXATRIACONTANE  
 $C_{36}H_{72}$

T. TRZEBIATOWSKI, M. DRÄGER and G.R. STROBL, 1982. Makromol. Chem., 183, 731-744.

$(CH_2)_{36}$

Monoclinic,  $Aa$ ,  $a = 10.33$ ,  $b = 8.24$ ,  $c = 42.2 \text{ \AA}$ ,  $\beta = 107.1^\circ$ ,  $Z = 4$ .  $R = 0.25$  for 839 reflexions (at  $-160^\circ\text{C}$ ).

The molecules consist of two stretched stems which are linked by sharp folds at each end (Fig. 1). The folds have the conformation  $t(gg\bar{t}gg)t$  lying parallel to the  $b$  axis. (No distances and angles have been supplied, presumably because of the low accuracy in structure determination.)

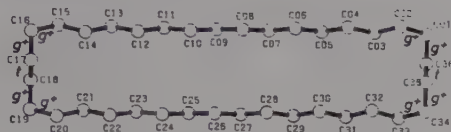


Fig. 1. Molecular structure of  $C_{36}H_{72}$  showing conformation.

#### OCTABROMONAPHTHALENE

$C_{10}Br_8$

J.H. BRADY, A.D. REDHOUSE and B.J. WAKEFIELD, 1982. J. Chem. Research, S, 137; M, 1541-1554.

Orthorhombic,  $Pbcn$ ,  $a = 11.47$ ,  $b = 7.94$ ,  $c = 16.69 \text{ \AA}$ ,  $Z = 4$ . Mo radiation,  $R = 0.105$  for 310 reflexions.

The structure is distorted with large deviations from the mean plane (Fig. 1). The molecule has a twofold axis of symmetry.

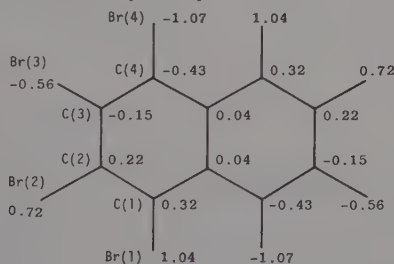


Fig. 1.  $C_{10}Br_8$ : deviations from the best least-squares plane.

#### 2-NAPHTHYSULPHONYLHYDRAZINE

$C_{10}H_{10}N_2O_2S$

G.V. GRIDUNOVA, V.E. SHKLOVER, Yu.T. STRUCHKOV and B.A. CHAYANOV, 1982. Cryst. Struct. Comm., 11, 1879-1884.

Monoclinic,  $P2_1/c$ ,  $a = 10.700$ ,  $b = 8.5998$ ,  $c = 11.2767 \text{ \AA}$ ,  $\beta = 104.81^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.050$  for 1243 reflexions.

The naphthalene nucleus (Fig. 1) is slightly non-planar (dihedral angle between the aromatic rings  $2.0^\circ$ ). Dimensions of the naphthyl moiety are as expected. Molecules are linked by N-H...O hydrogen bonds ( $N...O 3.000(4) \text{ \AA}$ ) about inversion centres.

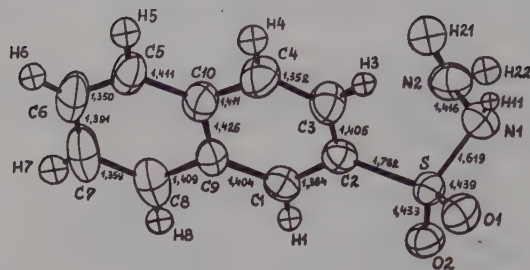
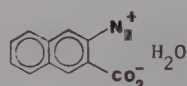


Fig. 1. A perspective view of  $C_{10}H_{10}N_2O_2S$  ( $\sigma$  0.002-0.008 Å).

# NAPHTHALENE-2-DIAZONIUM-3-CARBOXYLATE MONOHYDRATE

$C_{11}H_6N_2O_2 \cdot H_2O$

J.Z. GOUGOUTAS, 1982. Cryst. Struct. Comm., 11, 1305-1311.



Triclinic,  $P\bar{1}$ ,  $a = 6.650$ ,  $b = 10.436$ ,  $c = 8.533$  Å,  $\alpha = 106.71^\circ$ ,  $\beta = 119.46^\circ$ ,  $\gamma = 94.39^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.04$  for 1620 reflexions (at  $-85^\circ C$ ).

The somewhat bent diazonium group ( $C-N-N$   $172.3(2)^\circ$ ) and carboxyl carbon are displaced to opposite sides of the aromatic ring plane. The carboxyl group is rotated  $7^\circ$  out of the aromatic plane.

# 1,4-DIHYDRONAPHTHOIC ACID

$C_{11}H_{10}O_2$

A.K. CHEETHAM, M.C. GROSSEL and D. JAMES, 1982. J. Org. Chem., 47, 566-568.

Monoclinic,  $P2_1/c$ ,  $a = 5.566$ ,  $b = 6.146$ ,  $c = 29.170$  Å,  $\beta = 86.48^\circ$ ,  $D_m = 1.2$ ,  $Z = 4$ . Mo radiation,  $R = 0.048$  for 1370 reflexions.

The conformation of this acid is described as a 'flattened boat', Fig. 1, with the interplanar angle,  $\alpha$ , of  $169.2^\circ$ . The carbonyl substituent is the pseudoaxial position.

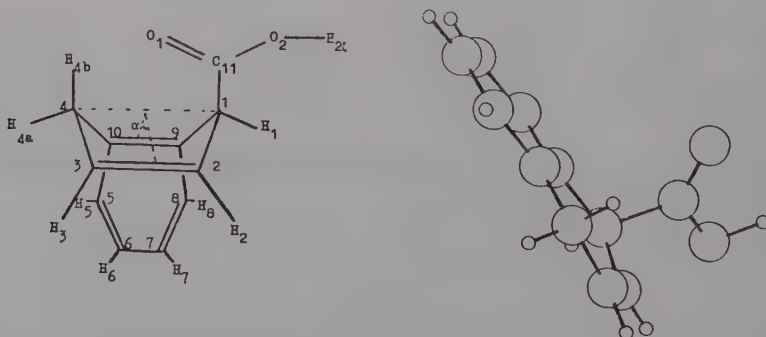


Fig. 1. Formula and view of 1,4-dihydronaphthoic acid.

3,5-EPOXY-2-METHYL-1,2,3,4-TETRAHYDRO-1-NAPHTHOL  
 $C_{11}H_{12}O_2$

J.P. GLUSKER, D.E. ZACHARIAS, D.L. WHALEN, S. FRIEDMAN and T.M. POHL, 1982. *Science*, **215**, 695-696.

Triclinic,  $P\bar{1}$ ,  $a = 8.098$ ,  $b = 9.938$ ,  $c = 6.279$  Å,  $\alpha = 93.93$ ,  $\beta = 104.35$ ,  $\gamma = 113.60^\circ$ . Mo radiation,  $R = 0.091$  for 1276 reflexions (at  $-80^\circ\text{C}$ ).

The structure determination reveals an intramolecular hydrogen bond  $O(1)\cdots H(O1)\cdots O(2)$  with  $O(1)\cdots O(2)$  2.792 Å and  $O-H\cdots O$   $142^\circ$  (Fig. 1). The epoxide ring is approximately symmetrical with no distortion due to steric effects, and the two ring substituents (the OH on C(1) and the methyl group on C(2)) occupy axial positions.

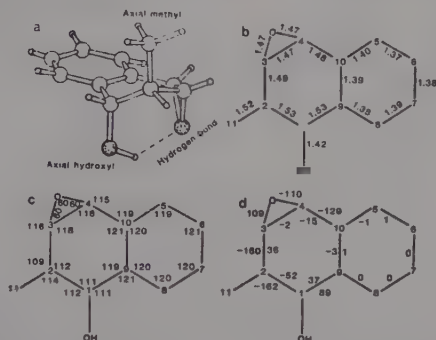


Fig. 1. General view of the molecule  $C_{11}H_{12}O_2$  showing the intramolecular hydrogen bond (a), bond lengths (b), bond angles (c), and torsion angles (d).

2,3,4-TRICHLORO-1-NAPHTHALENEGLYOXYLIC ACID  
 $C_{12}H_5Cl_3O_3$

J.S. CANTRELL, J.L. PYLE, P.A. LONG and R.A. LUNSFORD, 1982. *Acta Cryst.*, **B38**, 996-998.

Monoclinic,  $P2_1/a$ ,  $a = 17.199$ ,  $b = 6.094$ ,  $c = 12.057$  Å,  $\beta = 106.10^\circ$ ,  $D_m = 1.681$ ,  $Z = 4$ . Mo radiation,  $R = 0.053$  for 1848 reflexions.

In the molecule (Fig. 1) the roughly planar glyoxylic acid group is rotated  $60^\circ$  out of the naphthalene plane. The compound crystallizes as hydrogen-bonded dimers between carboxylic acid groups about a centre of symmetry. The  $\alpha$ -CO does not participate in the hydrogen bonding in this crystal structure.



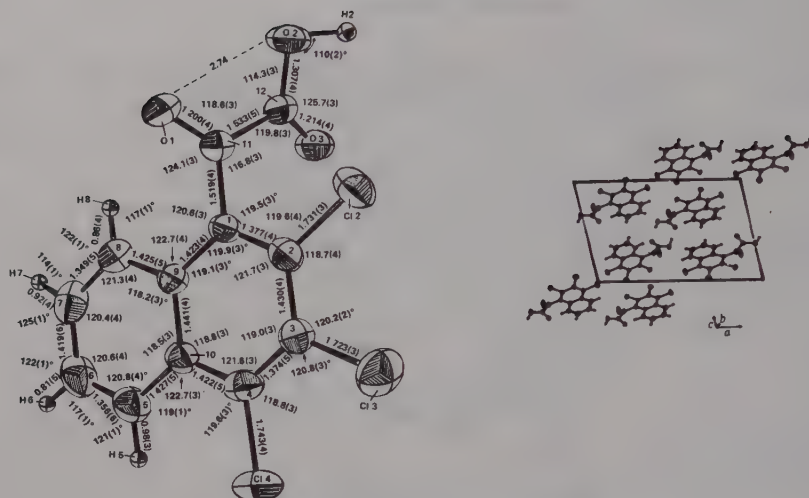


Fig. 1.  $C_{12}H_5Cl_3O_3$ : bond lengths and angles and a view of the crystal structure down the b axis.

$\omega$ -BROMO-2-ACETONAPHTHALENE  
 $C_{12}H_9BrO$

K.N. PRASAD, 1982. Ind. J. Pure Appl. Phys., 20, 412-413.

Orthorhombic,  $Pna2_1$ ,  $a = 20.650$ ,  $b = 4.731$ ,  $c = 10.398$  Å,  $D_m = 1.597$ ,  $Z = 4$ . Cu radiation,  $R = 0.072$  for 514 reflexions.

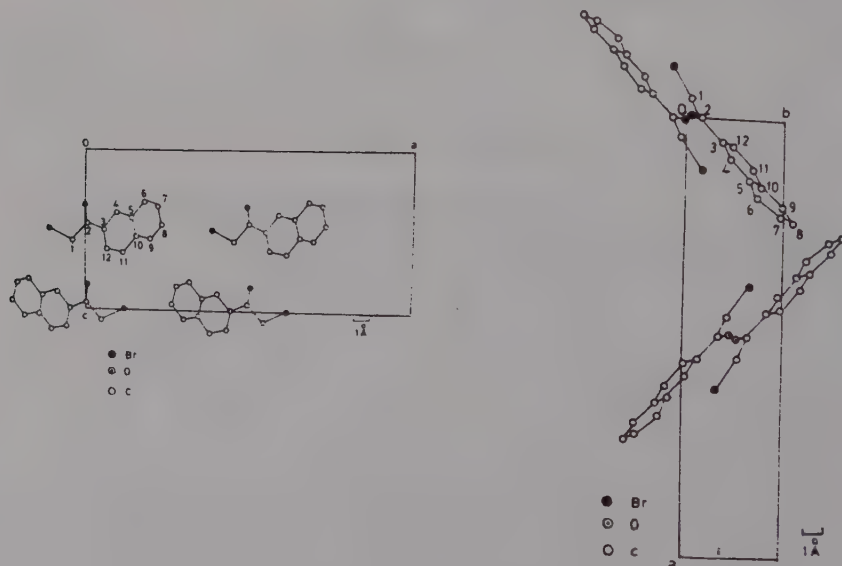


Fig. 1. Projections of the crystal structure of  $C_{12}H_9BrO$  along the b-axis (left), and the c-axis (right).

The naphthalene moiety is approximately planar (Fig. 1) with a maximum deviation from the plane through the ten C atoms of  $-0.116 \text{ \AA}$  for C(6). The side chain is almost coplanar, with deviations of  $+0.242$ ,  $+0.119$ ,  $+0.022$  and  $+0.687 \text{ \AA}$  from the naphthalene plane for atoms C(1), C(2), O and Br respectively. The Br-C(1) bond length is  $1.93 \text{ \AA}$ , C(1)-C(2)  $1.53$ , C(2)-O  $1.24$  and C(2)-C(3)  $1.57 \text{ \AA}$ . The bond lengths within the naphthalene rings range from  $1.37$  to  $1.42 \text{ \AA}$ .

# 1,8-DIMORPHOLINONAPHTHALENE

$\text{C}_{18}\text{H}_{22}\text{N}_2\text{O}_2$  (I)

# 1,8-DIPIPERIDINONAPHTHALENE

$\text{C}_{20}\text{H}_{26}\text{N}_2$  (II)

W. WONG-NG, S.C. NYBURG, A. AWWAL, R. JANKIE and A.J. KRESGE, 1982. *Acta Cryst.* B38, 559-564.

I. Monoclinic,  $P2_1/c$ ,  $a = 11.086$ ,  $b = 9.870$ ,  $c = 16.378 \text{ \AA}$ ,  $\beta = 121.2^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.059$  for 1508 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 9.151$ ,  $b = 9.380$ ,  $c = 10.118 \text{ \AA}$ ,  $\alpha = 105.9$ ,  $\beta = 98.8$ ,  $\gamma = 94.2^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.066$  for 1956 reflexions.

In both molecules (Fig. 1) the naphthalene ring system is distorted, the more so in II than I, as shown by the angles (in I)  $\text{N}(1)\text{-C}(1)\text{-C}(4)$   $173.1^\circ$ ,  $\text{N}(2)\text{-C}(8)\text{-C}(5)$   $173.8^\circ$ ,  $\text{N}(1)\text{-C}(1)\text{-C}(8)\text{-N}(2)$   $-18.97^\circ$ ,  $\text{C}(1)\text{-C}(9)\text{-C}(10)\text{-C}(4)$   $7.76^\circ$  and  $\text{C}(8)\text{-C}(9)\text{-C}(10)\text{-C}(5)$   $8.79^\circ$ . The corresponding angles in II are  $172.4$ ,  $173.3$ ,  $-21.61$ ,  $9.77$  and  $10.2^\circ$ . Both molecules have a pseudo diad axis about C(9)-C(10) with maximum deviations  $0.039 \text{ \AA}$  (I) and  $0.034 \text{ \AA}$  (II). The sum of the angles at N are  $337.1$  and  $337.6^\circ$  (I) and  $340.1$  and  $337.9^\circ$  (II).

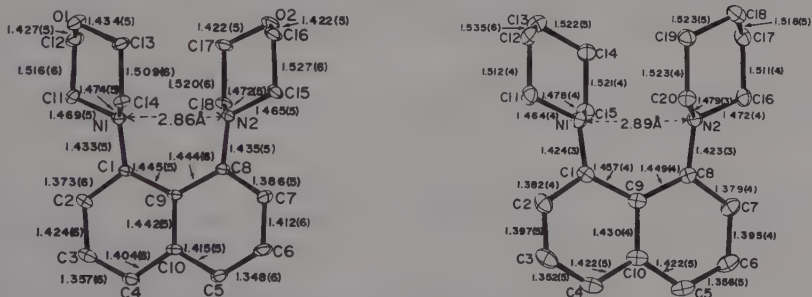


Fig. 1. Views of  $\text{C}_{18}\text{H}_{22}\text{N}_2\text{O}_2$  (I) and  $\text{C}_{20}\text{H}_{26}\text{N}_2$  (II).

# OCTAMETHYLNAPHTHALENE

$\text{C}_{18}\text{H}_{14}$

G.A. SIM, 1982. *Acta Cryst.*, B38, 623-625.

Orthorhombic,  $Ccca$ ,  $a = 16.595$ ,  $b = 11.242$ ,  $c = 7.632 \text{ \AA}$ ,  $D_m = 1.089$ ,  $Z = 4$ . Cu radiation,  $R = 0.045$  for 478 reflexions.

The molecule (Fig. 1) has  $D_2$  symmetry. Successive pairs of  $\alpha$  and  $\beta$  methyl carbon atoms are displaced alternatively above and below the mean molecular plane with buckling of the naphthalene nucleus. The  $\text{C}(1)\text{-C}(9)\text{-C}(8)$  angle is  $124.3^\circ$  (about  $3^\circ$  greater than in naphthalene). These distortions lead to methyl C...C separations of about  $2.9 \text{ \AA}$  and only one H...H contact is less than  $2 \text{ \AA}$ . Bond lengths in the rings are similar to those in naphthalene. For previous work see (1).

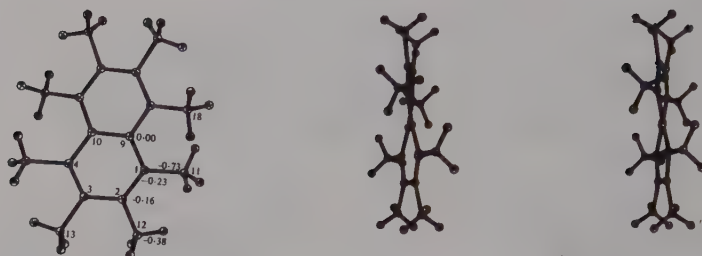


Fig. 1. The octamethylnaphthalene molecule with deviations (Å) of atoms from the mean molecular plane, and a stereoview.

1. Structure Reports, 17, 732.

# 2-(4-CYCLOHEXYLNAPHTHALENE)PROPIONIC ACID

$C_{19}H_{22}O_2$

L. DUPONT, O. DIDEBERG, G. DIVE, J.J. GODFROID and E. STEINER, 1982. Acta Cryst., B38, 2409-2411.

Triclinic,  $P\bar{1}$ ,  $a = 10.918$ ,  $b = 13.279$ ,  $c = 6.241$  Å,  $\alpha = 97.13$ ,  $\beta = 89.05$ ,  $\gamma = 60.71^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.079$  for 2612 reflexions.

In the molecule (Fig. 1) the rotation of the carboxyl group with respect to the benzene ring, which seems to be connected with anti-inflammatory potential, is larger than in other 2-substituted propionic acids already reported. Bond lengths and angles are generally as expected. In the crystal the molecules are linked into pairs across a centre of symmetry by hydrogen bonds with  $O \dots O$  2.659(2) Å.

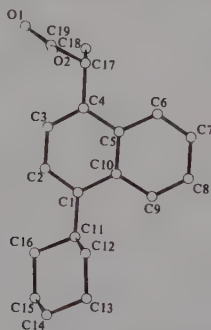


Fig. 1.  $C_{19}H_{22}O_2$ : perspective view of the molecule.

# 2,2'-BIS(1-NAPHTHOL)

$C_{20}H_{14}O_2$  (I)

$(C_{10}H_6OH)_2$

# 2,2'-BIS(1-AMINONAPHTHALENE)

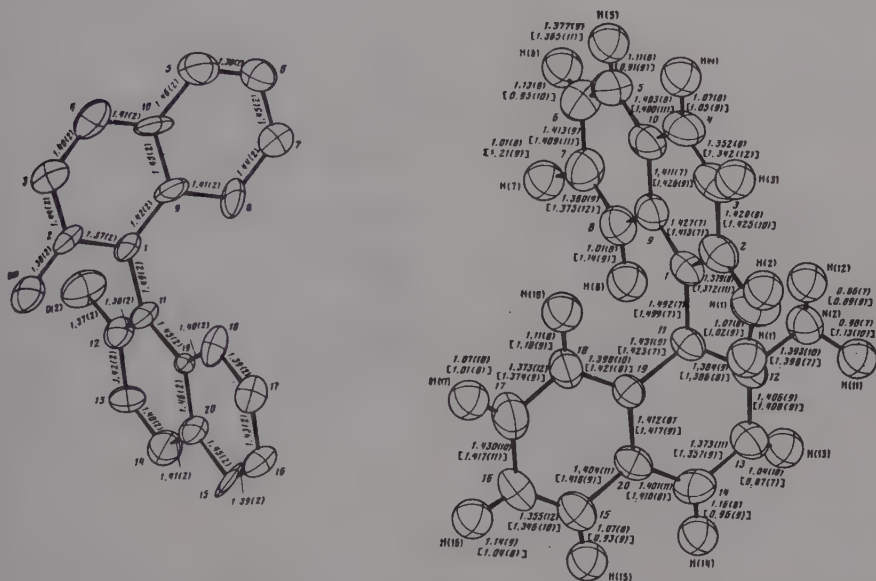
$C_{20}H_{16}N_2$  (II)

$(C_{10}H_6NH_2)_2$

G.V. GRIDUNOVA, N.G. FURMANOVA, V.E. SHKLOVER, Yu.T. STRUCHKOV, Z.I. EZHKOVA and B.A. CHAYANOV, 1982. Kristallografiya, 27, 477-484 [Sov. Phys. Crystallogr., 27, 290-294].

I. Orthorhombic,  $Iba2$ ,  $a = 15.691$ ,  $b = 21.617$ ,  $c = 8.628$  Å,  $Z = 8$ . Mo radiation,  $R = 0.085$  for 680 reflexions.

The crystals of I and II are pseudoisomorphous (Fig. 1) and in II there are two independent molecules per asymmetric unit. The dihedral angle between the planes of the naphthyl nuclei is  $89^\circ$  in I and  $85^\circ$  in II. Molecules of I are linked by hydrogen bonds ( $\text{O-H}\cdots\text{O}$  2.84 Å) formed about the  $2_1$  axes through OH groups. A similar arrangement exists in the structure of II, between the  $\text{-NH}_2$  groups ( $\text{N}\cdots\text{N}$  3.164 and 3.13 Å).



E-3-(3'-METHYLTHIO-1'-CARBOMETHOXYPROPYLIDINYL)-5 $\beta$ -AMINOMETHYL-8a-PHENYLMETHYL-trans-PERHYDRONAPHTHALENE  
C<sub>24</sub>H<sub>35</sub>NO<sub>2</sub>S

Triclinic,  $P1$ ,  $a = 11.029$ ,  $b = 13.460$ ,  $c = 8.709$  Å,  $\alpha = 97.80$ ,  $\beta = 110.34$ ,  $\gamma = 68.78^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.077$  for 2624 reflexions.

In this structure (Fig. 1) the rigid nature of the two cyclohexane rings with chair conformations in the trans-perhydronaphthalene system, forces the functional groups on the periphery into specific conformations. Atoms C20 and C22 are pushed away from each other; the C3-C11-C12-O13 torsion angle is  $37^\circ$  so that ester cannot fully conjugate with the C3-C11 double bond.

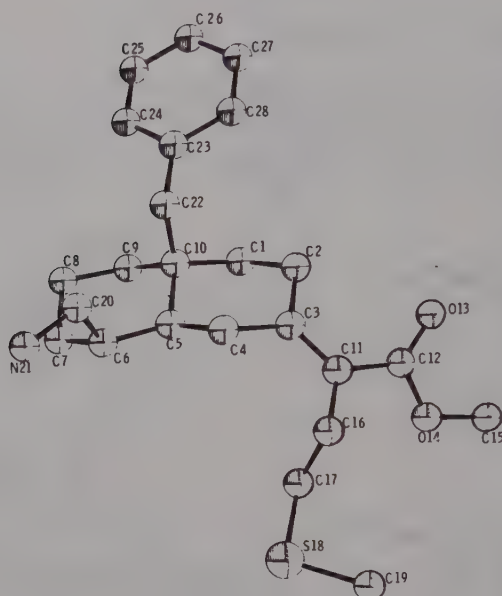


Fig. 1. A view of the  $C_{24}H_{35}NO_2S$  E-isomer.

# 1,4,5,8-NAPHTHALENETETRONE



F.H. HERBSTEIN and M. KAPON, 1982. Acta Cryst., B38, 3123-3125.

Monoclinic,  $P1_1/n$ ,  $a = 6.510$ ,  $b = 13.220$ ,  $c = 4.855 \text{ \AA}$ ,  $\beta = 110.0^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.07$  for 1102 reflexions.

The molecule (Fig. 1) lies on an inversion centre but has almost exact 2/m symmetry with the twofold axis along C(9)-C(10); the carbonyl oxygens are displaced by  $\pm 0.265 \text{ \AA}$  from the plane through the central six C atoms.

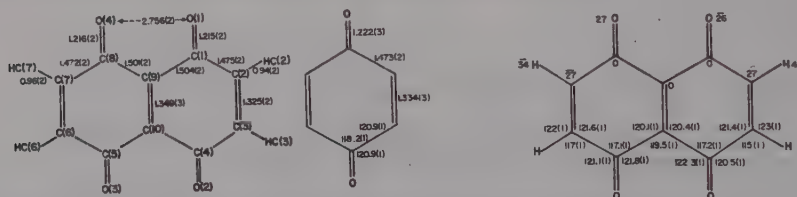
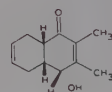
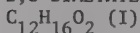
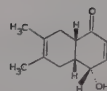


Fig. 1. Bond lengths and angles for  $C_{10}H_4O_4$ .

# 2,3-DIMETHYL-4 $\alpha$ ,5,8,8 $\alpha$ -TETRAHYDRO-1-NAPHTHOQUIN-4 $\alpha$ -OL



6,7-DIMETHYL-4 $\alpha$ ,5,8,8 $\alpha$ -TETRAHYDRO-1-NAPHTHOQUIN-4 $\alpha$ -OL  
C<sub>12</sub>H<sub>16</sub>O<sub>2</sub> (II)



A.S. SECCO and J. TROTTER, 1982. Acta Cryst., B38, 1233-1237.

I. Orthorhombic, P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>,  $a = 5.148$ ,  $b = 12.269$ ,  $c = 16.478$  Å,  $Z = 4$ . Mo radiation,  $R = 0.031$  for 671 reflexions.

II. Monoclinic, P2<sub>1</sub>/c,  $a = 9.242$ ,  $b = 22.724$ ,  $c = 5.139$  Å,  $\beta = 102.7^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.036$  for 626 reflexions.

Molecules in the two crystals (Fig. 1) adopt a twisted conformation and are joined by O(4)-H...O(4') hydrogen bonds. All other intermolecular contacts correspond to normal van der Waals distances. Solid state photochemistry is explained on the basis of the structural data obtained.

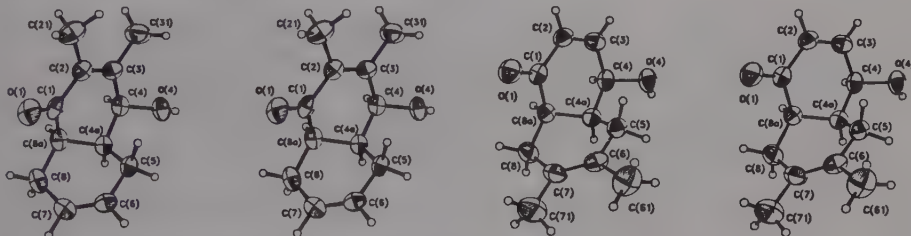
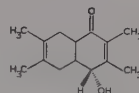


Fig. 1. Stereoscopic views of C<sub>12</sub>H<sub>16</sub>O<sub>2</sub> (I) (left) and C<sub>12</sub>H<sub>16</sub>O<sub>2</sub> (II) (right).

2,3,6,7-TETRAMETHYL-4 $\alpha$ ,5,8,8 $\alpha$ -TETRAHYDRO-1-NAPHTHOQUIN-4 $\alpha$ -OL  
C<sub>14</sub>H<sub>20</sub>O<sub>2</sub>



A.S. SECCO and J. TROTTER, 1982. Acta Cryst., B38, 1229-1232.

Monoclinic, C2/c,  $a = 13.898$ ,  $b = 5.228$ ,  $c = 17.316$  Å,  $\beta = 97.442^\circ$ ,  $D_m = 1.174$ ,  $Z = 4$ . Mo radiation,  $R = 0.058$  for 888 reflexions.

The structure (Fig. 1) contains molecules lying on twofold axes as a result of disorder within the lattice. Two types of hydrogen bonding, O(4)...O(1') (2.652 Å) and O(4)...O(4'') (2.804 Å), join molecules into a network within slabs parallel to (001). Molecules adopt a conformation where the hydroxyl group occupies a position pseudo-equatorial to the cyclohexenone ring.

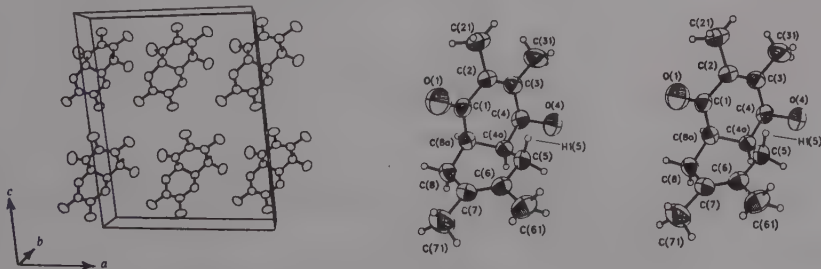


Fig. 1. C<sub>14</sub>H<sub>20</sub>O<sub>2</sub>: molecular packing viewed approximately down  $b$  (left) (only one set of the disordered molecules is shown), and a stereoscopic view of a molecule (right).



2,3,4 $\alpha$ ,4 $\alpha$  $\beta$ ,6,7,8 $\alpha\beta$ -HEPTAMETHYL-4 $\alpha\beta$ ,5,8,8 $\alpha\beta$ -TETRAHYDRO-1-NAPHTHOQUIN-4 $\beta$ -OL  
 $C_{17}H_{26}O_2$

A.S. SECCO and J. TROTTER, 1982. *Acta Cryst.*, **B38**, 2190-2196.

Monoclinic,  $P2_1/c$ ,  $a = 7.497$ ,  $b = 16.792$ ,  $c = 12.687$  Å,  $\beta = 105.30^\circ$ ,  $D_m = 1.140$ ,  $Z = 4$ . Mo radiation,  $R = 0.041$  for 1934 reflexions.

Despite the syn hydroxyl group, the bulkier methyl group causes the molecule (Fig. 1) to adopt a conformation typical of OH-anti derivatives, i.e. a half-chair cyclohexene ring cis-fused to a half-chair cyclohexenone moiety, with the 4-methyl substituent pseudo-equatorial. Bond distances and angles are generally normal. The molecules are linked in the crystal along a by  $O(4)-H...O(1)$  hydrogen bonds with  $O...O$  2.855(2) Å. This structure is compared with those of other tetrahydronaphthoquinol derivatives.

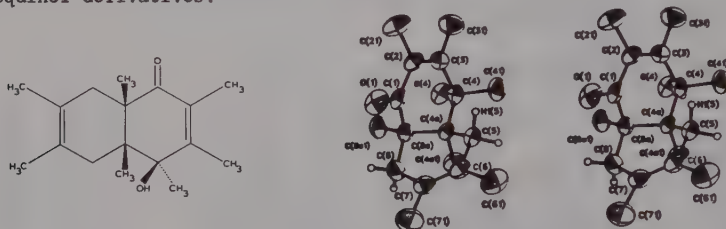


Fig. 1.  $C_{17}H_{26}O_2$ : structural formula and stereodiagram of the molecule.

ANTHRACENE- $d_{10}$

$C_{14}D_{10}$

S.L. CHAPLOT, N. LEHNER and G.S. PAWLEY, 1982. *Acta Cryst.*, **B38**, 483-487.

Monoclinic,  $P2_1/a$ ,  $a = 8.37$ ,  $b = 6.00$ ,  $c = 11.12$  Å,  $\beta = 125.4^\circ$ . Neutron radiation ( $\eta = 0.8567$  Å),  $R = 0.032$  for 1105 reflexions.

Anthracene- $d_{10}$  has been studied by neutron diffraction at room temperature (1). The present study reveals significant out-of-plane and in-plane shearing distortions in the crystal from the  $mmm$  symmetry of the free molecule. Thermal motion analysis shows that the T tensor is most reduced at low temperature with the thermal motion of the D nuclei hardly affected. A riding-motion correction of +0.014 Å is necessary for the C-D bond lengths in Fig. 1 giving an average C-D length of 1.106(1) Å.



Fig. 1. Bond lengths (Å) and angles ( $^\circ$ ) for  $C_{14}D_{10}$  after refinement with constrained  $mmm$  symmetry (e.s.d.s. 0.003 Å and  $0.1^\circ$ ).

1. Structure Reports, **38B**, 198.

## 9-BROMO-10-METHYLANTHRACENE



T.R. WELBERRY, R.D.G. JONES and J. EPSTEIN, 1982. *Acta Cryst.*, B38, 1518-1525.

Orthorhombic,  $P2_12_12_1$ ,  $a = 17.439$ ,  $b = 16.039$ ,  $c = 3.9590$  Å,  $Z = 4$ . Mo radiation,  $R = 0.051$  for 981 reflexions.

The 9, 10 substituents show disorder of Br and CH<sub>3</sub>-groups, the 9-substituent being occupied 0.613 Br, 0.387 CH<sub>3</sub> and vice versa for the 10-substituent. There is short range ordering between adjacent molecules when the contact involves a CH<sub>3</sub>- or Br in both molecules. There are two types of contact involved and for both, short-range order reduces Br...Br contacts in favour of Br...C. There is no evidence for split Br/methyl positions or disorder in the anthracene ring. Molecular geometry (Fig. 1) is normal with Br-C(9) and C(10)-C(15) lying between accepted Br-C(aromatic) and C(methyl)-C(aromatic) distances.

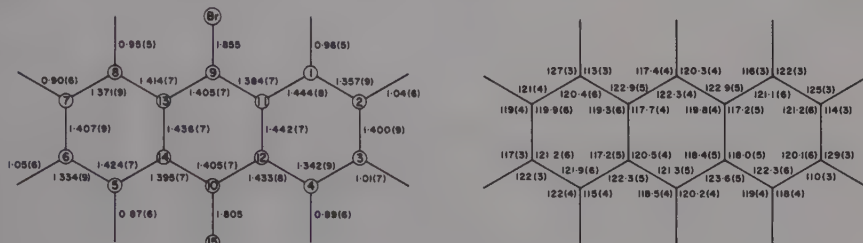
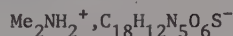
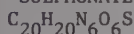


Fig. 1. Bond lengths and angles in 9-bromo-10-methylanthracene.

DIMETHYLAMMONIUM 1-AMINO-4-(3-ACETYLAMINO-1,2,4-TRIAZOL-1-YL)ANTHRAQUINONE-2-SULPHONATE



J.-M. ADAM, T. WINKLER and G. RIHS, 1982. *Helv. Chim. Acta*, 65, 2318-2325.

Monoclinic,  $P2_1/c$ ,  $a = 8.072$ ,  $b = 16.608$ ,  $c = 15.389$  Å,  $\beta = 96.59^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.051$  for 2462 reflexions.

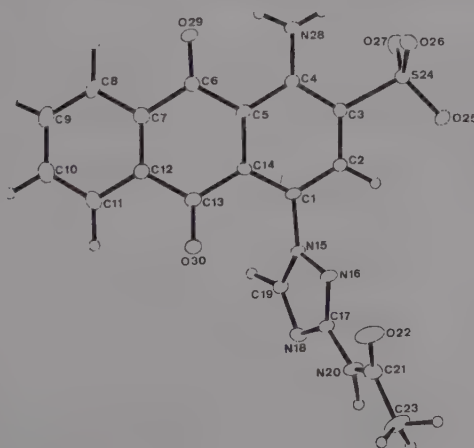
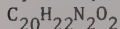


Fig. 1. A view of the  $C_{18}H_{12}N_5O_6S^-$  anion.

The analysis established the structure of this anthraquinone sulphononic acid derivative as shown in Fig. 1. The triazole ring plane is inclined at  $63^\circ$  to the C(1)-C(5),C14 plane; the two aromatic rings of the anthraquinone are  $15^\circ$  from planarity.

# 1,4-BIS(ISOPROPYLAMINO)ANTHRAQUINONE



C.J. BROWN and G.R. MITCHELL, 1982. *Acta Cryst.*, **B38**, 3149-3151.

Trigonal,  $R\bar{3}$ ,  $a = 15.030 \text{ \AA}$ ,  $\alpha = 106.4^\circ$ ,  $D_m = 1.165$ ,  $Z = 6$ .  $\text{MoK}\alpha$  radiation,  $R = 0.056$  for 2307 reflexions.

The structure (Fig. 1) comprises discrete molecules with two internal hydrogen bonds (N-H...O) per molecule, but no intermolecular interactions other than van der Waals forces.

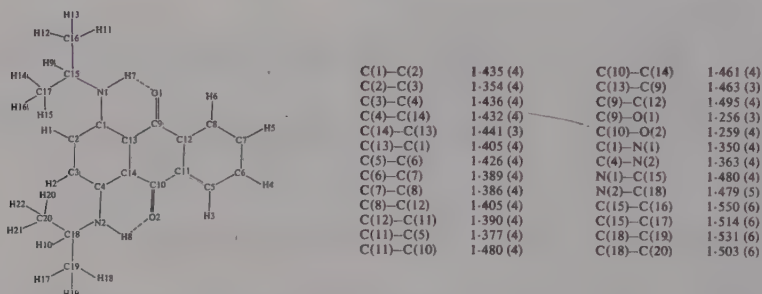


Fig. 1. A diagram of  $\text{C}_{20}\text{H}_{22}\text{N}_2\text{O}_2$  and bond lengths.

N-{2-[(2-CHLOROETHYL)THIO]ETHYL}-10-METHYL-9-ANTHRACENEMETHYLAMINE HYDROCHLORIDE  
 $\text{C}_{20}\text{H}_{23}\text{Cl}_2\text{NS}$  (I)

10-METHYL-9-[(4-THIOMORPHOLINO)METHYL]ANTHRACENE  
 $\text{C}_{20}\text{H}_{21}\text{NS}$  (II)

W.C. STALLINGS, C.T. MONTI and J.P. GLUSKER, 1982. *Acta Cryst.*, **B38**, 177-184.

I. Orthorhombic,  $P2_12_12_1$ ,  $a = 22.748$ ,  $b = 13.007$ ,  $c = 6.3443 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.050$  for 1220 reflexions.

II. Monoclinic,  $P2_1/a$ ,  $a = 35.816$ ,  $b = 6.8965$ ,  $c = 13.895 \text{ \AA}$ ,  $\beta = 105.89^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.047$  for 4513 reflexions.

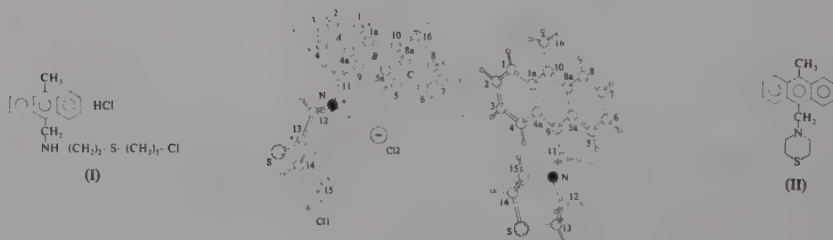
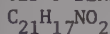


Fig. 1. Perspective views of  $\text{C}_{20}\text{H}_{23}\text{Cl}_2\text{NS}$  (I) (left) and  $\text{C}_{20}\text{H}_{21}\text{NS}$  (II) (right).

In compound I (Fig. 1) the atoms of the anthracene ring are coplanar and bond lengths approximate those predicted by simple resonance theory. The molecules are linked in the crystal through N-H...C1 hydrogen bonds. In compound II (Fig. 1) there are two independent molecules, with similar bond lengths and angles, in the asymmetric unit. The anthracene rings are not planar, and there is an angle between the outer rings of  $5.2^\circ$  (molecule A) and  $3.0^\circ$  (molecule B). In both structures the anthracene ring systems of the molecules do not overlap in planes  $3.4 \text{ \AA}$  apart, but they do lie in parallel planes, approximately  $3.5 \text{ \AA}$  apart, with a remarkably similar motif for each.

cis-9-BENZYL-10-NITRO-9,10-DIHYDROANTHRACENE



G. BARTOLI, M. BOSCO, R. DAL POZZO and P. SGARABOTTO, 1982. J. Chem. Soc. Perkin II, 929-932.

Orthorhombic,  $\text{Pca}2_1$ ,  $a = 14.650$ ,  $b = 14.622$ ,  $c = 7.623 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.039$  for 1034 reflexions.

The nitro and benzyl groups are in a boat axial conformation (Fig. 1), the nitro group making an angle of  $75.2^\circ$  to the mean plane of C(11)-C(14) with N(1)-C(9) at  $92.6^\circ$  to the C(11)...C(14) direction. The substituents do not appear to alter the geometry of the cis-9,10-dihydroanthracene. The dihedral angle between the mean planes of the two benzene rings is  $148.5^\circ$ , values of  $145$  to  $155^\circ$  being reported for related compounds. The dihedral angles between the C(11)-C(14) mean plane and the planes of C(9), C(11), C(14) and C(10), C(12), C(13) are  $25.5$  and  $26.0^\circ$  respectively. The crystal packing is determined by van der Waals forces.

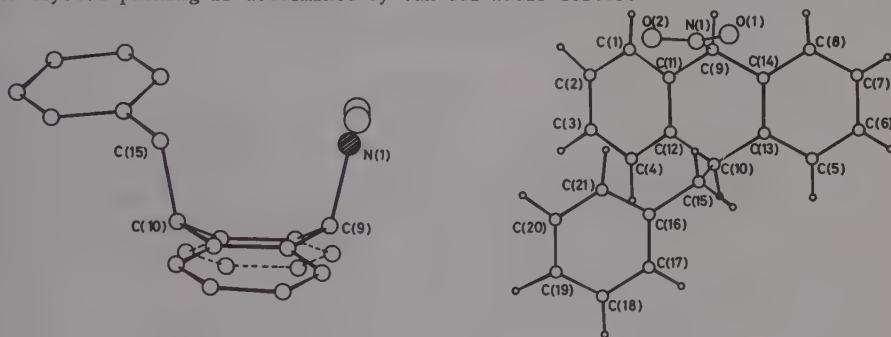
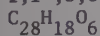


Fig. 1. Projection on to the C(11)-C(14) mean plane (left) and molecular structure of  $\text{C}_{21}\text{H}_{17}\text{NO}_2$  (right).

1,1',8,8'-TETRAHYDROXY-10,10'-BI-9(10H)-ANTHRONE (ANTHRALIN DIMER)



F.R. AHMED and G.A. NEVILLE, 1982. Acta Cryst., B38, 2930-2933.

Monoclinic,  $\text{C}2/c$ ,  $a = 17.708$ ,  $b = 7.566$ ,  $c = 16.263 \text{ \AA}$ ,  $\beta = 106.60^\circ$ ,  $D_m = 1.43$ ,  $Z = 4$ . Cu radiation,  $R = 0.041$  for 778 reflexions.

The molecule (Fig. 1) consists of two halves related by a crystallographic twofold axis normal to the central C-C bond linking the two anthracene moieties. This bond is stretched to a length of  $1.612(7) \text{ \AA}$ , and its nearest endocyclic angle is  $114.5(3)^\circ$  compared to  $117.7(3)^\circ$  in anthralin. The three O atoms of each half are interlinked by intramolecular O-H...O...H-O hydrogen bonds. This crystal structure has already been reported elsewhere (1).

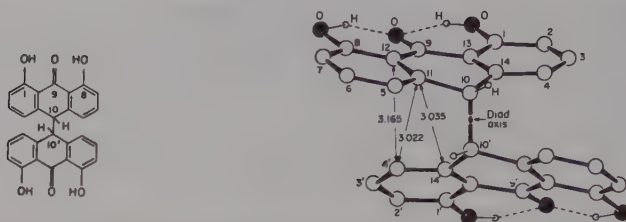


Fig. 1.  $C_{28}H_{18}O_6$ : molecular structure and perspective view of the molecule along the diad axis which relates the two equivalent halves.

1. Following Report.

1,1',8,8'-TETRAHYDROXY-10,10'-BI-9(10H)-ANTHRONE  
 $C_{28}H_{18}O_6$

M. WHITEFIELD, K. HENRICK and P.G. OWSTON, 1982. *Acta Cryst.*, B38, 1248-1251.

Monoclinic,  $C2/c$ ,  $a = 17.751$ ,  $b = 7.581$ ,  $c = 16.316$  Å,  $\beta = 106.64^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.066$  for 846 reflexions. [See also the preceding Report.]

There is a crystallographic twofold symmetry axis perpendicular to the C(10)-C(10') bond which links the two halves of the molecule (Fig. 1), and each half is markedly less planar than the monomeric precursor, 1,8-dihydroxy-9(10H)-anthrone (1). The C(10)-C(10') bond is long (1.62 Å) and probably weaker than a normal bond.

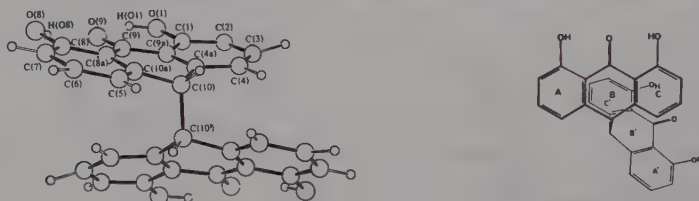


Fig. 1.  $C_{28}H_{18}O_6$ : a perspective view of the molecule.

1. Structure Reports, 46B, 137.

1,2-BIS(10-ACETOXY-9-ANTHRYL)ETHANE ( $\alpha$ -PHASE)  
 $C_{34}H_{26}O_4$  (I)

1,2-BIS(10-ACETOXY-9-ANTHRYL)ETHANE ( $\beta$ -PHASE)  
 $C_{34}H_{26}O_4$  (II)

H.-D. BECKER, S.R. HALL, B.W. SKELTON and A.H. WHITE, 1982. *Aust. J. Chem.*, 35, 2357-2365.

I. Monoclinic,  $P2_1/c$ ,  $a = 7.609$ ,  $b = 14.469$ ,  $c = 11.358$  Å,  $\beta = 96.39^\circ$ ,  $D_m = 1.33$ ,  $Z = 2$ . Mo radiation,  $R = 0.034$  for 1341 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 31.264$ ,  $b = 5.248$ ,  $c = 15.718$  Å,  $\beta = 97.84^\circ$ ,  $D_m = 1.30$ ,  $Z = 4$ . Mo radiation,  $R = 0.052$  for 972 reflexions.

Both structures contain centrosymmetric molecules. In I (Fig. 1) molecules are stacked along the  $a$ -axis with a 3.75 Å interplanar separation. In II the separation is 3.44 Å and the stacking is along the  $b$ -axis.

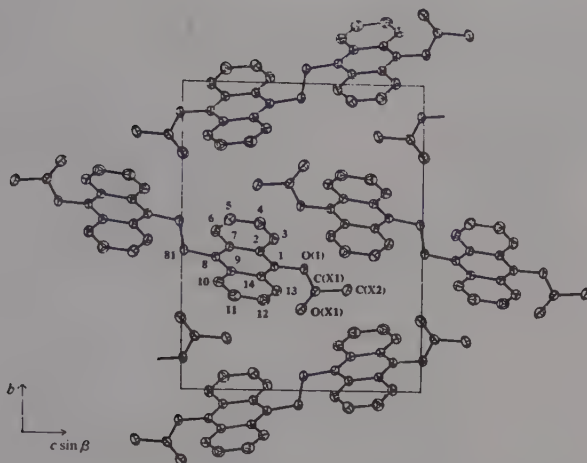


Fig. 1. The unit cell contents of the  $\alpha$ -phase of  $C_{34}H_{26}O_4$  (I) projected down  $a$ .

(2S,3S)-2-exo-BROMO-3-endo-HYDROXYBICYCLO[3.2.0]HEPTAN-6-ONE  
 $C_7H_9BrO_2$

P. MURRAY-RUST, R.C. GLEN and R.F. NEWTON, 1982. *Acta Cryst.*, **B38**, 2700-2702.

Trigonal,  $P3_1$ ,  $a = 10.13$ ,  $c = 6.66$  Å,  $D_m = 1.72$ ,  $Z = 3$ . Mo radiation,  $R = 0.0416$  for 563 reflexions.

In the molecule (Fig. 1) the cyclopentane ring is in the envelope conformation and the hydroxy group is pseudo-axial; the absolute chirality has been determined. There is evidence for a weak intramolecular  $O \cdots C=O$  interaction, with  $O(3) \cdots C(6)$  2.990(15) Å. The molecules are linked in the crystal by hydrogen bonds along the  $3_1$  axis, with  $O(3) \cdots O(3')$  2.766(15) Å.

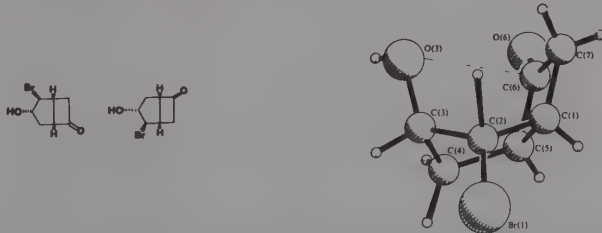


Fig. 1.  $C_7H_9BrO_2$ : the conformation and absolute chirality of the molecule with the transannular  $O \cdots C=O$  interaction shown as a dotted line.

2-HYDROXYHOMOTROPYLIUM HEXACHLOROANTIMONATE(V)  
 $C_8H_9Cl_6OSb$

$[C_8H_8OH]^+ [SbCl_6]^-$

R.F. CHILDS, A. VARADARAJAN, C.J.L. LOCK, R. FAGGIANI, C.A. FYFE and R.E. WASYLISHEN, 1982. *J. Am. Chem. Soc.*, **104**, 2452-2456.

Monoclinic,  $A2/m$ ,  $a = 8.893$ ,  $b = 11.093$ ,  $c = 18.811$  Å,  $\beta = 128.32^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.045$  for 1936 reflexions (at  $-65^\circ C$ ).

The cation (Fig. 1) is disordered, but the refinement is sufficiently good to



say that the internal cyclopropane bond is longer than normal (1.626(8) Å) and the other cyclopropane bonds shorter than normal (1.488(7) Å). These bond distances are fully in accord with what would be expected for a homoaromatic cation.



Fig. 1. The  $C_8H_8(OH)^+$  cation, dihedral angles between planes of atoms shown at right.

2-DIISOPROPYLAMINO-3,7-DEHYDROTROPONE  
 $C_9H_{17}NO$

R. GRIEB, R. W. GRAY and A.S. DREIDING, 1982. *Helv. Chim. Acta*, **65**, 1045-1049.

Monoclinic,  $P2_1/b$ ,  $a = 13.68$ ,  $b = 14.51$ ,  $c = 6.687$  Å,  $\gamma = 114.3^\circ$ ,  $Z = 4$ . [?] radiation,  $R = 0.049$  for 631 reflexions.

The analysis confirms the structure shown in Fig. 1, with all ring C atoms trigonal and planar.

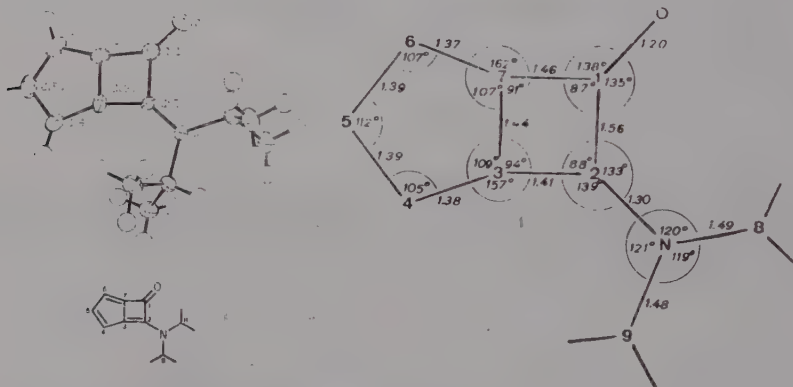


Fig. 1. The  $C_9H_{17}NO$  molecule and dimensions.

(1RS,5RS,7SR)-1-ACETOXY-7-CYANO-5-METHYLBICYCLO[3.2.0]HEPTAN-2-ONE  
 $C_{11}H_{13}NO_3$

A. FURUSAKI, K. ABE, T. MATSUMOTO, 1982. *Bull. Chem. Soc. Jpn.*, **55**, 611-612.

Monoclinic,  $P2_1$ ,  $a = 13.852$ ,  $b = 6.750$ ,  $c = 13.096$  Å,  $\beta = 118.69^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.044$  for 2229 reflexions.

The two crystallographically independent molecules in the asymmetric unit have opposite absolute configurations and similar geometries (Fig. 1). The cyclopentanone ring has half-chair form. The cyclobutane ring is puckered (puckering angle  $12^\circ$ ).

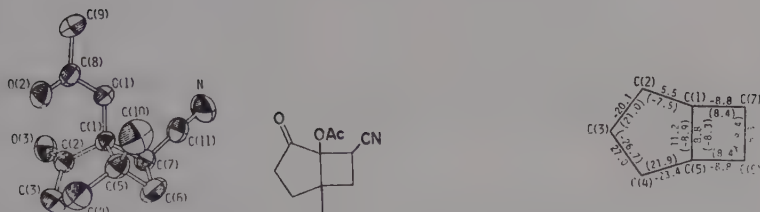


Fig. 1. A  $C_{11}H_{13}NO_3$  molecule and torsion angles (values in parentheses refer to the second molecule).

3 $\beta$ -METHYL-cis-HEXAHYDRO-4-INDANONE SEMICARBAZONE

$C_{11}H_{19}N_3O$  (I)

3 $\beta$ -METHYL-cis-HEXAHYDRO-4-INDANONE THIOSEMICARBAZONE

$C_{11}H_{19}N_3S$  (II)

3 $\beta$ ,7 $\alpha$ -DIMETHYL-cis-HEXAHYDRO-4-INDANONE SEMICARBAZONE

$C_{12}H_{21}N_3O$  (III)

Y. JEANNIN, C. STORA, F. ROBERT, F. WEISBUCH, G. DANA and B. LOCICERO, 1982. *Acta Cryst.*, B38, 1773-1777.

I. Monoclinic,  $P2_1/c$ ,  $a = 12.270$ ,  $b = 7.060$ ,  $c = 13.717$  Å,  $\beta = 96.38^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.062$  for 1894 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 12.153$ ,  $b = 8.499$ ,  $c = 12.068$  Å,  $\beta = 93.85^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.062$  for 1727 reflexions.

III. Monoclinic,  $B2_1/c$ ,  $a = 13.416$ ,  $b = 7.263$ ,  $c = 26.185$  Å,  $\beta = 96.40^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.057$  for 2094 reflexions.

The three molecules (Fig. 1) have the 3 $\beta$ -methyl-cis-hydrindan-4-one configuration with H(C3a) cis with respect to H(C7a) or C(10). The cyclohexane rings have chair and cyclopentane envelope conformations. Bond lengths are similar in the three molecules except for C(7a)-C(1) which is 1.485(6) (I), 1.528(7) (II) and 1.557(4) Å (III), and C(2)-C(3) which is abnormally long (1.585 Å) in I.

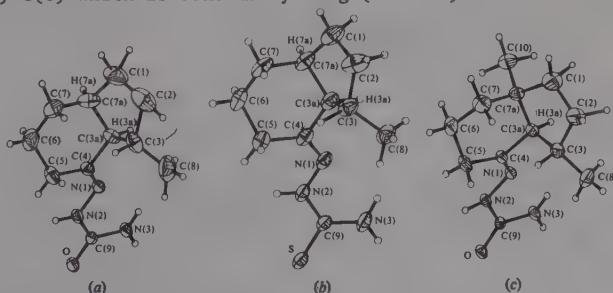


Fig. 1. The molecules of (a)  $C_{11}H_{19}N_3O$  (I), (b)  $C_{11}H_{19}N_3S$  (II), and (c)  $C_{12}H_{21}N_3O$  (III).

trans-6,10-DIMETHYLBICYCLO[4.4.0]DEC-1-ENE-3 $\alpha$ -CARBONITRILE

$C_{13}H_{19}N$

B. BEAGLEY, P. MORTON, R.G. PRITCHARD and R. RAMAGE, 1982. *Acta Cryst.*, B38, 1393-1395.

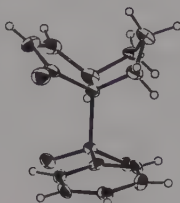
The figure contains two ORTEP diagrams, labeled (I) and (II). Diagram (I) shows the molecular structure of compound (I), which is a tricyclic molecule with a central six-membered ring fused to two five-membered rings. It features several oxygen atoms (O1, O2, O3) and carbon atoms (C1-C10). A small chemical structure of the molecule is shown below the ORTEP diagram. Diagram (II) shows the molecular structure of compound (II), which is a tricyclic molecule with a central six-membered ring fused to two five-membered rings. It features several oxygen atoms (O1, O2, O3) and carbon atoms (C1-C13). A small chemical structure of the molecule is shown below the ORTEP diagram.

Fig. 1. The  $C_{13}H_{20}O_3$  (I) and  $C_{13}H_{18}O_3$  (II) molecules.

## 1-PHENYLTHIOBICYCLO[3.3.0]OCTA-3-EN-2-ONE

 $C_{14}H_{14}O_3S$ P.A. MAGNUS, D.A. QUAGLIATO and J.C. HUFFMAN, 1982. *Organometallics*, **1**, 1240-1242.Triclinic,  $P1$ ,  $a = 10.358$ ,  $b = 12.981$ ,  $c = 9.323$  Å,  $\alpha = 89.41^\circ$ ,  $\beta = 100.55^\circ$ ,  $\gamma = 90.03^\circ$ ,  $Z = 4$ .  $R = 0.077$  for 2893 observed reflexions (at  $-165^\circ\text{C}$ ).

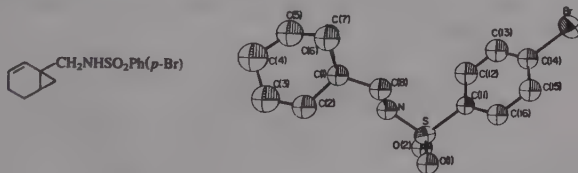
The structure of this reaction product is shown in Fig. 1. [No geometrical details are published, fractional coordinates are available.] The molecular packing is reported to be pseudomonoclinic ( $P2_1/n$ ).

Fig. 1. A view of the  $C_{14}H_{14}O_3S$  molecule.

## BICYCLO[4.1.0]HEPT-2-EN-1-YLMETHANAMINE p-BROMOBENZENESULFONAMIDE

 $C_{14}H_{16}BrNO_2S$ I.A. AKHTAR, J.J. McCULLOUGH, S. VAITEKUNAS, R. FAGGIANI and C.J.L. LOCK, 1982. *Canad. J. Chem.*, **60**, 1657-1663.Monoclinic,  $P2_1/c$ ,  $a = 13.551$ ,  $b = 5.993$ ,  $c = 18.832$  Å,  $\beta = 105.44^\circ$ ,  $D_m = 1.50$ ,  $Z = 4$ . Mo radiation,  $R = 0.081$  for 1004 reflexions.

The structure of this reaction intermediate is shown in Fig. 1. Molecular geometry details are in accord with accepted values. Atom C5 is 0.768(9) Å from the C1-C4 plane compared with 0.340(7) Å for C6 and -1.065(9) Å for C7. The dihedral angle between the cyclopropane ring (C1,C6,C7) and C5,C6,C1,C2 is  $108.1(2)^\circ$ .

Fig. 1. The  $C_{14}H_{16}BrNO_2S$  molecule.

## 1-ALLYL-3,3-DIMETHYL-8-METHYLBICYCLO[3.3.0]OCTANE-2,7-DIONE

 $C_{14}H_{22}O_2$ R.D. ADAMS, 1982. *Cryst. Struct. Comm.*, **11**, 547-550.Monoclinic,  $P2_1/c$ ,  $a = 15.037$ ,  $b = 6.530$ ,  $c = 14.814$  Å,  $\beta = 114.23^\circ$ .  $Z = 4$ . Mo radiation,  $R = 0.070$  for 1045 reflexions.

The stereochemistry of the molecule (Fig. 1) is such that allyl addition has occurred at the bridgehead carbon of the carbon-carbon double bond and, as expected, has resulted in cis-relationship of the substituents on the bridgehead carbons. The presence of the methyl substituent on carbon C5 allows one to establish that the stereochemistry of the addition of the allyl group and hydrogen atom to the double bond in the precursor is trans. The C12-C13 double bond of the allyl group is

disordered, approximately equally, between two rotational conformations.

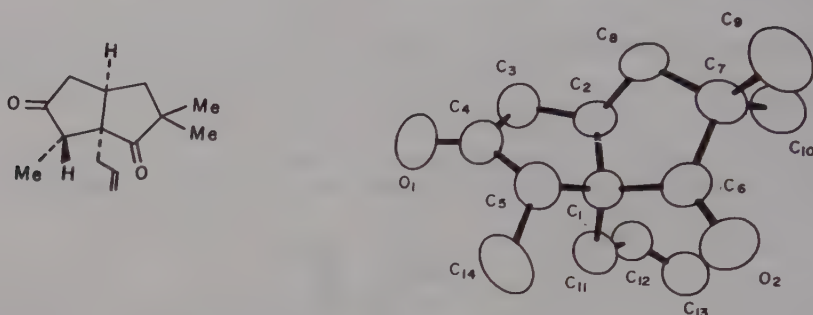


Fig. 1. The C<sub>14</sub>H<sub>22</sub>O<sub>2</sub> molecule.

r-1,t-2,c-3,t-4,5-PENTACHLORO-8-PHENYLTETRALIN  
C<sub>16</sub>H<sub>11</sub>Cl<sub>5</sub>

M.C. AVERSA, G. BOMBIERI, G. BRUNO, G. CUM, P. GIANNETTO and G. ROMEO, 1982.  
J. Chem. Soc. Perkin II, 1213-1216.

Monoclinic, P2<sub>1</sub>/c, a = 9.600, b = 13.647, c = 12.132 Å, β = 90.1°, Z = 4. Mo radiation, R = 0.044 for 1075 reflexions.

The two aromatic rings (Fig. 1) are planar within experimental error, and the angle between the mean plane of the fused rings and the C(11)-C(16) ring is 61.6°. The C(8)-C(11) bond length (1.49 Å) corresponds to a C(sp<sup>2</sup>)-C(sp<sup>2</sup>) single bond indicating markedly reduced conjugation between the non-coplanar aromatic rings. The Cl(5)-C(5) bond distance (1.734) is longer than normal for chlorobenzenes (1.69 Å), also indicating reduced double bond character. The mean C-C bond length in the C(11)-C(16) ring is 1.38 Å, the shortening probably being due to libration effects. C(2) and C(3) are displaced respectively 0.170 above and 0.213 Å below the C(1)-C(10) mean plane, resulting in a rather flattened half-chair conformation for the puckered ring, which reduces the mutual repulsion between the axial and pseudo-axial Cl pairs (Cl(2)...Cl(4) 3.282 and Cl(1)...Cl(3) 3.347 Å).

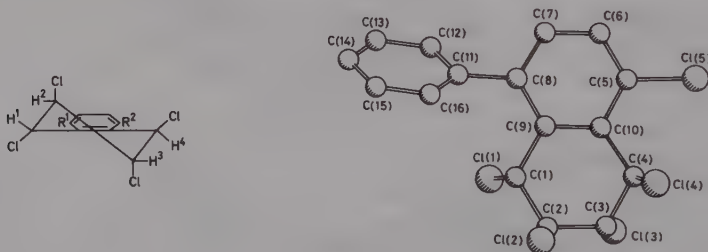


Fig. 1. Molecular skeleton (R<sup>1</sup> = Ph, R<sup>2</sup> = Cl) and structure of C<sub>16</sub>H<sub>11</sub>Cl<sub>5</sub>.

5,7-DIHYDROXY-4-METHOXYCARBONYL-3-PHENYL-1-INDANONE METHANOL SOLVATE  
C<sub>17</sub>H<sub>14</sub>O<sub>5</sub>·CH<sub>4</sub>O

S.H. BERTZ, G. RIHS and R.B. WOODWARD, 1982. Tetrahedron, 38, 63-67.

Triclinic, P $\bar{1}$ , a = 8.150, b = 10.694, c = 11.432 Å, α = 121.32, β = 78.35, γ = 107.74°, Z = 2. Mo radiation, R = 0.044 for 2606 reflexions.

The analysis established the structure shown in Fig. 1. The indanone system is

nearly planar. The five-membered ring is in an envelope conformation although the degree of puckering is small. There is an intramolecular hydrogen bond O(14)-H...O(11). The methanol of solvation is hydrogen bonded to O(15)-H.

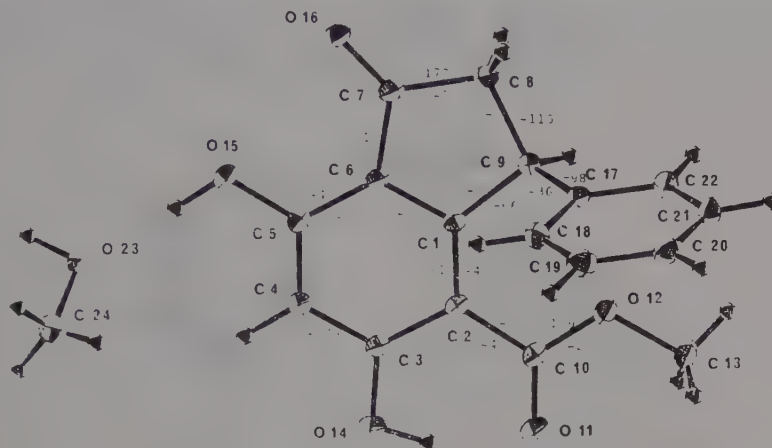


Fig. 1. A view of  $C_{17}H_{14}O_5 \cdot CH_4O$  with torsion angles.

rac-3,3'-BIINDAN-1-ONE

$$\text{C}_{18}\text{H}_{14}\text{O}_2$$

N.E. HEIMER, M. HOJJATIE and C.A. PANETTA, 1982. *J. Org. Chem.*, 47, 2593-2598.

Orthorhombic,  $P2_12_12$ ,  $a = 5.745$ ,  $b = 19.313$ ,  $c = 6.040$  Å,  $Z = 4$ . Mo radiation,  $R = 0.055$  for 1062 reflexions.

The analysis established the structure and stereochemistry shown in Fig. 1, with a skew conformation.

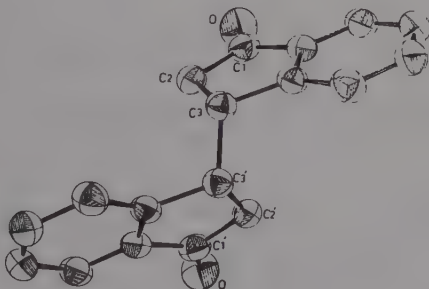


Fig. 1. The  $C_{18}H_{14}O_2$  molecule.

endo-4,4,6-TRIMETHYL-1,5-DIPHENYL-2,3-DIAZABICYCLO[3.1.0]HEX-2-ENE

$$\text{C}_{19}\text{H}_{20}\text{N}_2$$

K.M. BEEM, B. RUBIN, L.M. WAYKOLE and J.O. BRANNEN, 1982. *Cryst. Struct. Comm.*, **11**, 601-605.

Monoclinic, C2/c,  $a = 19.000$ ,  $b = 9.368$ ,  $c = 22.231$  Å,  $\beta = 124.77^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.055$  for 1282 reflexions.



The five-membered ring (Fig. 1) is puckered and the cyclopropyl ring is nearly equilateral with C-C 1.515(15) Å.

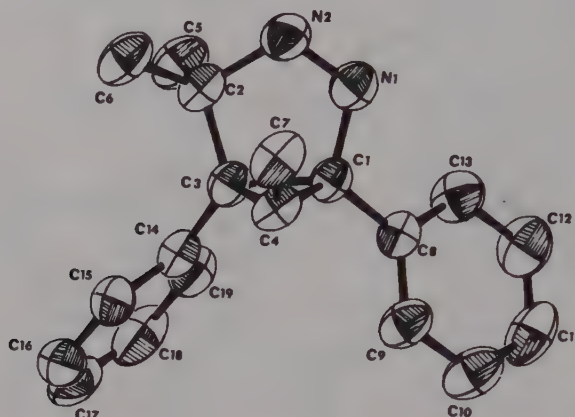


Fig. 1. A view of  $C_{19}H_{20}N_2$ .

trans-2 $\beta$ -METHYL-2 $\alpha$ -HYDROXY-5 $\beta$ -ISOPROPYL-7 $\beta$ -PHENYL-THIO-BICYCLO[4,3,0]-NONAN-8-ONE  
 $C_{19}H_{26}O_2S$

H. DEUTSCH and D. VANDERVEER, 1982. Cryst. Struct. Comm., 11, 445-450.

Triclinic,  $P1$ ,  $a = 7.009$ ,  $b = 9.256$ ,  $c = 14.60$  Å,  $\alpha = 99.90$ ,  $\beta = 110.09$ ,  $\gamma = 89.99^\circ$ ,  $D_m = 1.21$ ,  $Z = 2$ . Mo radiation,  $R = 0.085$  for 2280 reflexions.

The analysis shows the structure to be as shown in Fig. 1. The two independent molecules have the same stereochemistry.

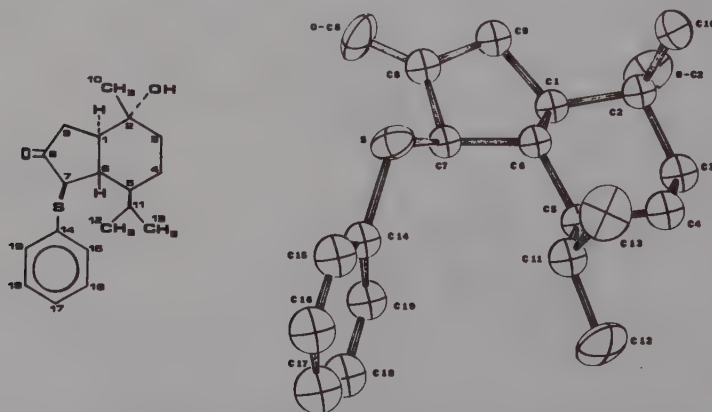


Fig. 1. The  $C_{19}H_{26}O_2S$  molecule and an Ortep plot of one of the molecules.

2-HYDROXYIMINO-3,3-DIPHENYLINDAN-1-ONE  
 $C_{21}H_{15}NO_2$

S. BRÜCKNER and L. MALPEZZI, 1982. Cryst. Struct. Comm., 11, 533-538.

Monoclinic,  $P2_1/c$ ,  $a = 12.779$ ,  $b = 10.069$ ,  $c = 26.340$  Å,  $\beta = 103.9^\circ$ ,  $[Z = 8]$ . Mo radiation,  $R = 0.046$  for 2147 reflexions.

The two independent molecules have anti-stereochemistry (Fig. 1) and the molecules are linked by O-H...O and O-H...N hydrogen bonds. The five-membered rings have shallow envelope conformations with C(2) at the flap. The C(1)-C(5) bond length is short 1.455(8) Å (average) and the adjacent C=O bond has C(1)-O(1) 1.226(11) Å (average).

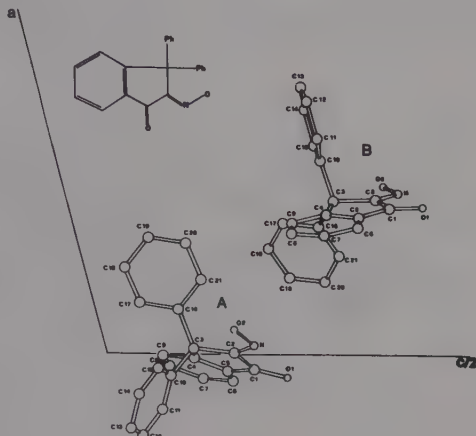


Fig. 1. The  $C_{21}H_{15}NO_2$  skeleton and the two independent molecules.

1R\*,5R\*,2'R\*-1-METHYL-2-METHYLENE-5-[2'-(1'-METHYLENE-6'-METHOXY-1',2',3',4'-TETRA-HYDRONAPHTHYL)]-1-CYCLOPENTANEACETALDEHYDE

$C_{21}H_{26}O_2$

R.D. ADAMS, H. LIM and F.E. ZIEGLER, 1982. Cryst. Struct. Comm., 11, 575-578.

Monoclinic,  $P2_1/n$ ,  $a = 11.759$ ,  $b = 7.010$ ,  $c = 21.362$  Å,  $\beta = 97.81^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.054$  for 1503 reflexions.

The aromatic ring (Fig. 1) is planar and the methoxyl group is twisted out of this plane by  $8^\circ$ . The cyclohexene ring has a chair conformation; the cyclopentane ring is an envelope.

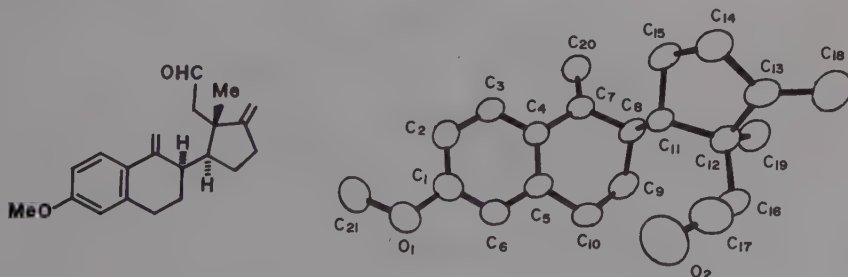
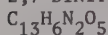


Fig. 1. The  $C_{21}H_{26}O_2$  molecule.

## 2,7-DINITROFLUORENONE



R.G. BAUGHMAN, 1982. Cryst. Struct. Comm., 11, 479-484.

Orthorhombic,  $\text{Pca}_2$ ,  $a = 20.998$ ,  $b = 3.793$ ,  $c = 14.252 \text{ \AA}$ ,  $Z = 4$ . Mo radiation,  $R = 0.068$  for 707 reflexions.

The nitro groups in the molecule (Fig. 1) are rotated out of the relevant ring planes by  $11^\circ$  (N(1)) and  $4^\circ$  (N(2)). There are numerous close contacts of oxygens with hydrogens of adjacent molecules.

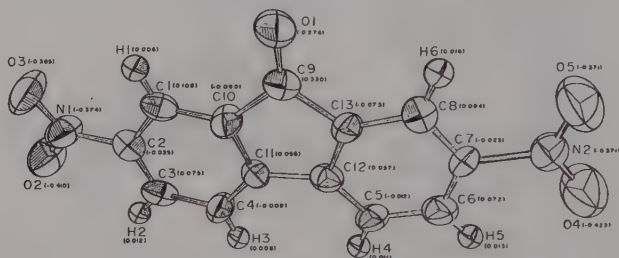


Fig. 1. A view of the 2,7-dinitrofluorenone molecule.

## 1-HYDROXY-9-FLUORENONE



T.L. BROWN, L.G. BUTLER, D.Y. CURTIN, Y. HIYAMA, I.C. PAUL and R.B. WILSON, 1982. J. Am. Chem. Soc., 104, 1172-1177.

Orthorhombic,  $\text{P2}_1\text{2}_1\text{2}_1$ ,  $a = 11.672$ ,  $b = 15.409$ ,  $c = 5.203 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.030$  for 865 reflexions.

The carbon framework of the molecule (Fig. 1) is planar to within  $\pm 0.025 \text{ \AA}$ ; O2 lies in the plane, but O1 and H01 are displaced by 0.071 and 0.247  $\text{ \AA}$ . H01 participates in an intramolecular (H...O 2.35(4)  $\text{ \AA}$ ) and an intermolecular (H...O 2.05(3)  $\text{ \AA}$ ) hydrogen bond with O2. Bond lengths ( $\sigma = 0.003\text{-}0.004 \text{ \AA}$ ) are as expected.

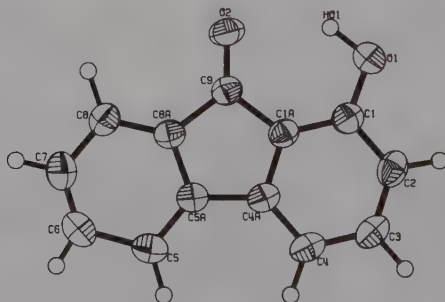


Fig. 1. The 1-hydroxy-9-fluorenone molecule.

## 2-(N-HYDROXYACETYLAMINO)FLUORENE



A. MICHEL, G. EVRARD and F. DURANT, 1982. Bull. Soc. Chim. Belg., 91, 341-342.

Monoclinic,  $P2_1$ ,  $a = 15.998$ ,  $b = 6.611$ ,  $c = 5.543 \text{ \AA}$ ,  $\beta = 95.81^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.049$  for 1061 reflexions.

Molecular dimensions are given in Fig. 1. The dihedral angle between the fluorene plane and that of the N-hydroxyacetyl amino moiety is  $14.7^\circ$ .

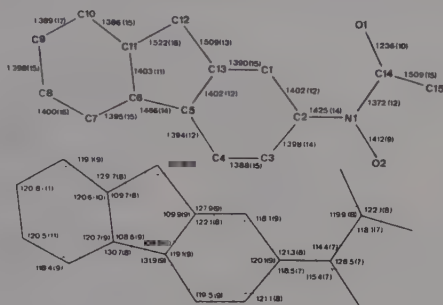


Fig. 1. Distances and angles in  $C_{15}H_{13}NO_2$ .

## 9-ACETOXY-10-BROMO-1,2,3,4,9,10,10a-OCTAHYDROPHENANTHRENE (BROMOHYDRIN ACETATE)



J.M. SAYER, H. YAGI, J.V. SILVERTON, S.L. FRIEDMAN, D.L. WHALEN and D.M. JERINA, 1982. J. Am. Chem. Soc., 104, 1972-1978.

Triclinic,  $P\bar{1}$ ,  $a = 8.3331$ ,  $b = 9.2842$ ,  $c = 10.3490 \text{ \AA}$ ,  $\alpha = 107.120$ ,  $\beta = 98.266$ ,  $\gamma = 101.525^\circ$ ,  $Z = 2$ .  $R = 0.054$ .

The structure analysis confirms the trans fusion of rings A and B (Fig. 1) and shows that O(11) lies on the same side of the ring as H(10a). The molecular dimensions ( $\sigma = 0.005$ - $0.014 \text{ \AA}$  and  $0.5$ - $0.7^\circ$ ) present no unusual features.

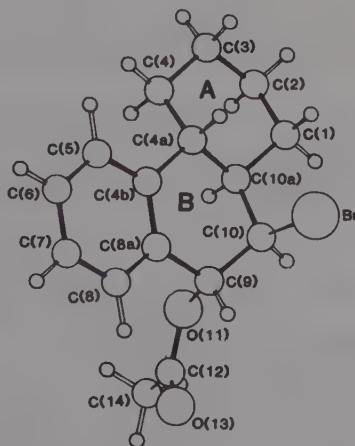


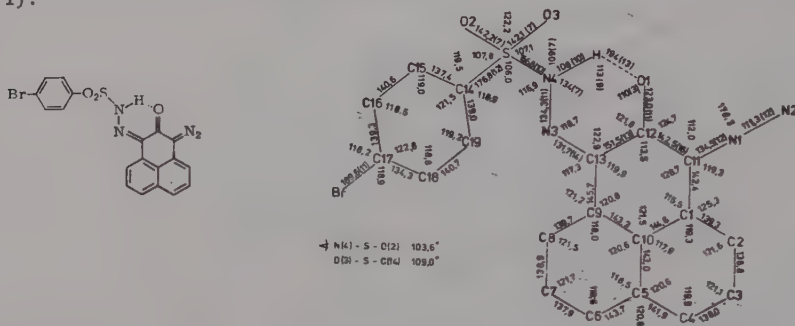
Fig. 1. The  $C_{16}H_{19}BrO_2$  molecule.

1-(4-BROMOPHENYLSULFONYLHYDRAZONO)-3-DIAZO-1H-PHENALEN-2(3H)-ONE  
 $C_{19}H_{11}BrN_4O_3S$

G. MAAS, O. GANSTER, M. REGITZ and B. EISTERT, 1982. Chem. Ber., 115, 435-443.

Triclinic,  $P\bar{1}$ ,  $a = 14.062$ ,  $b = 8.131$ ,  $c = 7.954$  Å,  $\alpha = 89.69$ ,  $\beta = 101.11$ ,  $\gamma = 100.79^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.042$  for 1090 reflexions.

The structure was confirmed as that shown in Fig. 1. The syn-orientation at the C(13)-N(3) double bond makes possible an intramolecular N-H...O hydrogen bond (Fig. 1).



(±)-(1α,3αβ,4αα,6e,8a)-OCTAHYDRO-6-((4-BROMOPHENYL)-METHYLENE)-1,4,4-TRIMETHYL-CYCLOPENTA(1,4)CYCLOBUTA(1,2)BENZEN-5(6H)-ONE

$C_{21}H_{25}BrO$

T.R. HOYE, S.J. MARTIN and D.R. PECK, 1982. J. Org. Chem., **47**, 331-337.

Monoclinic,  $P2_1/n$ ,  $a = 7.218$ ,  $b = 16.904$ ,  $c = 15.118$  Å,  $\beta = 100.89^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.042$  for 1584 reflexions.

Two of the products of the cycloaddition reactions in the irradiation of 3-(1,5-dimethylhex-4-enyl)cyclohex-2-enone have been identified by X-ray analysis of crystalline derivatives. The derivative of the major product had been shown (1) to have structure I (Fig. 1), and the current work corrects the proposed structure of the second product; its derivative has the structure II. Dimensions from the two analyses are compared; in particular, the differences in angles about C(1) where the methyl group (R/R') results from either exo or endo addition (in I and II respectively) were noted.

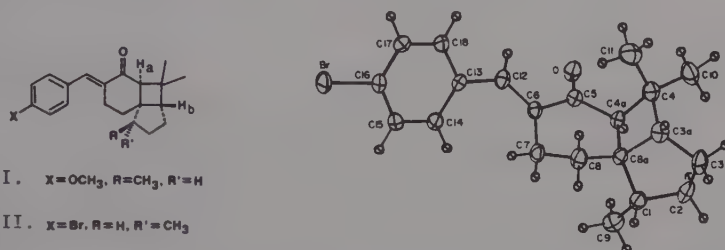
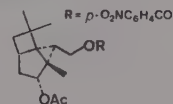


Fig. 1. Formulae for I and II, and a view of a molecule of II,  $C_{21}H_{25}BrO$ .

# 1. Structure Reports, 45B, 157.

(4-ACETOXY-3,8,8-TRIMETHYLTRICYCLO[4.2.0.0<sup>1,3</sup>]OCT-2-YL)METHYL-p-NITROBENZOATE

$C_{21}H_{25}NO_6$



K. ISHII, T.A. LYLE, W.B. SCHWEIZER and B. FREI, 1982. Helv. Chim. Acta, **65**, 595-599.

Monoclinic,  $P2_1/a$ ,  $a = 13.787$ ,  $b = 7.204$ ,  $c = 20.638$  Å,  $\beta = 101.83^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.039$  for 1854 reflexions.

The analysis shows that this reaction product has the structure shown above.

4,10-DIOXO-4αβ-CHLORO-5-(BENZYLOXY)-9β-HYDROXY-9α-METHYL-1,4,9,9α,10-PENTAHYDRO-ANTHRACENE

$C_{22}H_{19}ClO_4$

B.-M.G. GAVEBY, J.C. HUFFMAN and P. MAGNUS, 1982. J. Org. Chem., **47**, 3779-3780.

Monoclinic,  $P2_1/c$ ,  $a = 11.124$ ,  $b = 11.105$ ,  $c = 14.438$  Å,  $\beta = 95.21^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.083$  for 1491 reflexions (at  $-165^\circ C$ ).

The analysis established the structure shown in Fig. 1.



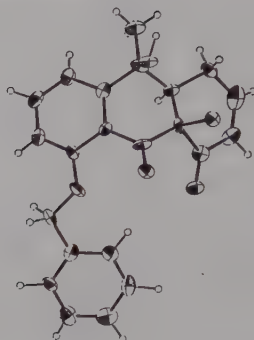
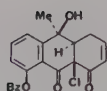
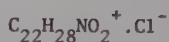
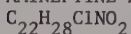


Fig. 1. The structure of  $C_{22}H_{19}ClO_4$ .

#### AMINEPTINE HYDROCHLORIDE



J.P. REBOUL, B. CRISTAU and G. PÈPE, 1982. *Acta Cryst.*, B38, 1489-1495.

Monoclinic,  $C2/c$ ,  $a = 22.456$ ,  $b = 7.673$ ,  $c = 26.328$  Å,  $\beta = 117.41^\circ$ ,  $D_m = 1.23$ ,  $Z = 8$ . Cu radiation,  $R = 0.066$  for 2971 reflexions.

The angle between the benzene ring planes is  $118.2(2)^\circ$ . The central ring has a boat form with C(5), C(6) and C(15) on the same side of the mean plane defined by C(11-14) (Fig. 1). The C(11)-C(15)-C(14) angle is  $114.8(2)^\circ$ . There is a Cl...H-O(25) hydrogen bond of length  $3.017(4)$  Å and several short Cl...N contacts in the range  $3.142$ - $3.281$  Å.

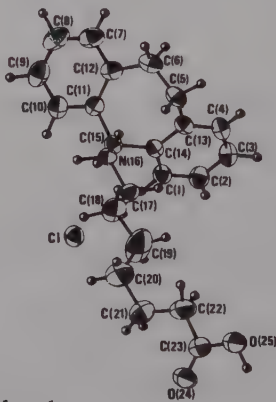
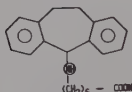


Fig. 1. The amineptine hydrochloride molecule.

#### trans,trans-TETRAKIS(METHOXYCARBONYL)DIBENZO[a,e]CYCLOOCTENE



D.W. JONES and W.S. McDONALD, 1982. *J. Chem. Soc. Perkin I*, 2257-2263.

Monoclinic,  $P2_1/c$ ,  $a = 8.712$ ,  $b = 15.544$ ,  $c = 16.122$  Å,  $\beta = 98.19^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.041$  for 2376 reflexions.

The eight-membered ring (Fig. 1) has a distorted tub conformation, with steric interaction between the axial substituents at C(5) and C(12) resulting in abnormally large ring angles at C(4a) and C(12a) of  $125.4^\circ$  (mean). The ring torsion angles

about C(5)-C(6) and C(11)-C(12) also deviate markedly from normal, the values for C(4a)-C(5)-C(6)-C(6a) and C(10a)-C(11)-C(12)-C(12a) being  $-8$  and  $-37^\circ$  respectively. Transannular non-bonded distances C(5)...C(12) and C(6)...C(11) are 3.161 and 2.915 Å respectively.

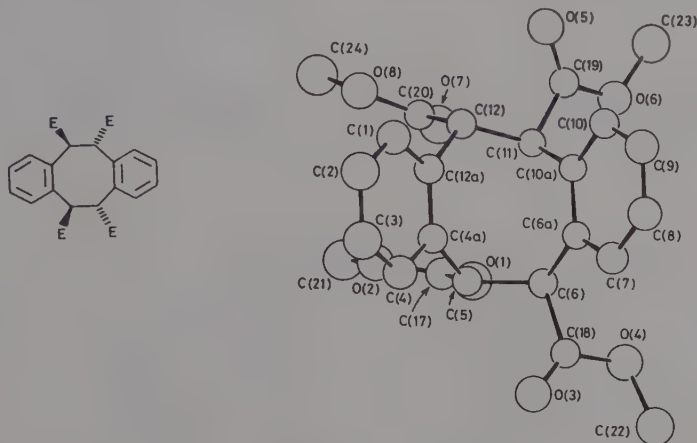


Fig. 1. Molecular skeleton and structure of  $C_{24}H_{24}O_8$ .

1 $\beta$ ,4 $\alpha\beta$ -DIMETHYL-7-METHOXY-1 $\alpha$ -(7,7'-ETHYLENEKETAL-6',6'-DIMETHYL-3'-KETOOCITYL)-3,4,4 $\alpha$ ,9,10,10 $\alpha$ -HEXAHYDRO-2(1H) PHENANTHRENE

$C_{29}H_{42}O_5$

J.W. APSIMON, R.P. SEQUIN and C.P. HUBER, 1982. *Canad. J. Chem.*, **60**, 509-513.

Triclinic,  $P\bar{1}$ ,  $a = 12.843$ ,  $b = 17.427$ ,  $c = 7.012$  Å,  $\alpha = 119.47$ ,  $\beta = 106.03$ ,  $\gamma = 85.37^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.051$  for 3824 reflexions.

The analysis shows this intermediate to have structure and dimensions given in Fig. 1. Ring A is planar, ring B is in the half-chair conformation, ring C is in a twist-boat form and the dioxolane ring is envelope shaped.

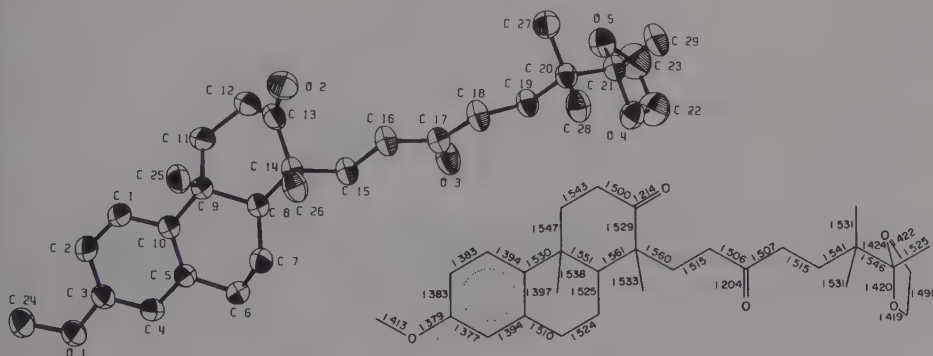
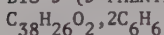


Fig. 1. A view of  $C_{29}H_{42}O_5$  and bond lengths (esd's are 0.002-0.004 Å).

## BIS-9-(9-PHENYL)FLUORENYL PEROXIDE BENZENE SOLVATE



T. PILATI, M. SIMONETTA and S. QUICI, 1982. *Cryst. Struct. Comm.*, **11**, 1027-1032.

Monoclinic,  $P2_1/n$ ,  $a = 18.979$ ,  $b = 9.728$ ,  $c = 10.092 \text{ \AA}$ ,  $\beta = 96.19^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.054$  for 1948 reflexions.

The molecule (Fig. 1) lies about an inversion centre, consequently the C-O-O-C torsion angle is  $180^\circ$ . Some dimensions are O-O  $1.491(3)$ , C-O  $1.441(2) \text{ \AA}$ ; C-O-O  $106.2(2)^\circ$ .

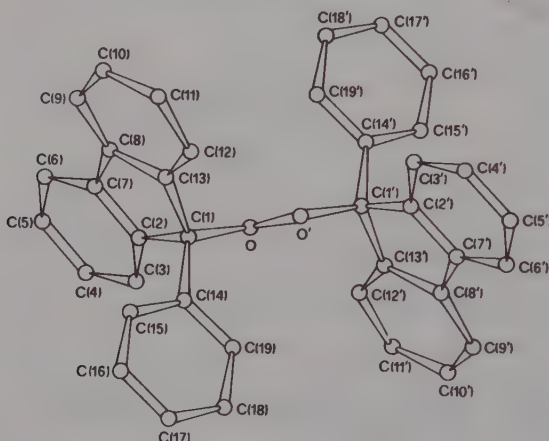
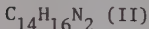


Fig. 1. A view of  $\text{C}_{38}\text{H}_{26}\text{O}_2$ .

cis-DICYANOTRIS[2.2.2]- $\sigma$ -HOMOBENZENEtrans-DICYANOTRIS[2.2.2]- $\sigma$ -HOMOBENZENE

G. McMULLEN, M. LUTTERBECK, H. FRITZ, H. PRINZBACH and C. KRÜGER, 1982. *Israel J. Chem.*, **22**, 19-26.

I. Orthorhombic,  $Pbca$ ,  $a = 11.1841$ ,  $b = 20.140$ ,  $c = 20.698 \text{ \AA}$ ,  $Z = 16$ . Cu radiation,  $R = 0.046$  for 2757 reflexions.

II. Monoclinic,  $P2_1/n$ ,  $a = 19.095$ ,  $b = 6.728$ ,  $c = 19.534 \text{ \AA}$ ,  $\beta = 106.23^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.096$  for 2622 reflexions.

Both compounds crystallize with two different conformers in the asymmetric units as shown in Fig. 1. The C-C bonds bearing the nitrile groups are elongated ( $1.58$ - $1.60 \text{ \AA}$ ). The average length of other bridging C-C bonds is  $1.554 \text{ \AA}$ , whereas non-bridging bonds are considerably shorter ( $1.519 \text{ \AA}$  average). In I, the six-membered ring is chair shaped whereas in II it approximates a twist boat. The dihedral within the twisted four-membered rings of all four molecules are similar (average  $17 \pm 3^\circ$ ).

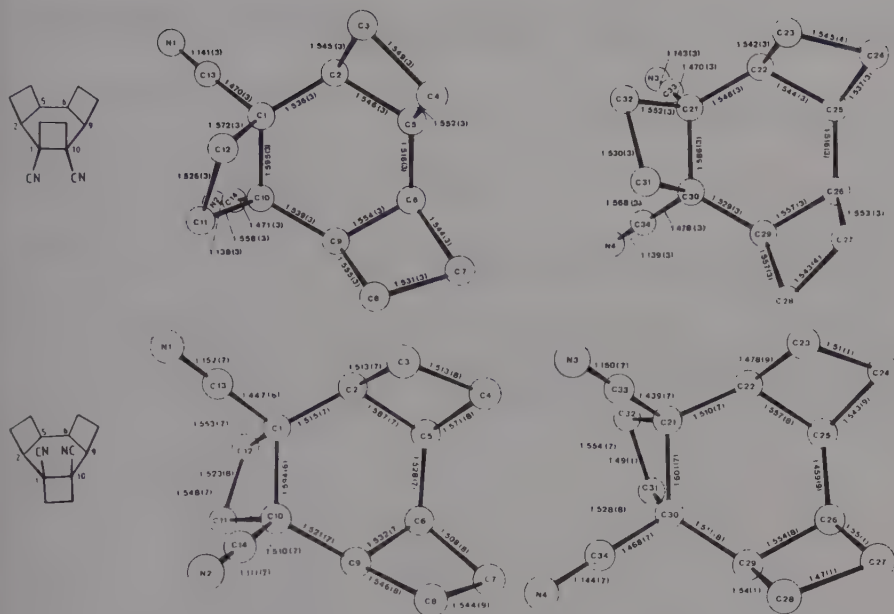


Fig. 1. The cis- (top) and trans- (bottom)  $C_{14}H_{16}N_2$  molecules.

# PERFLUOROTRIS(DIHYDROCYCLOPENTA) BENZENE

$C_{15}F_{18}$

F.W.B. EINSTEIN and T. JONES, 1982. *Acta Cryst.*, **B38**, 328-330.

Orthorhombic,  $Pbcn$ ,  $a = 17.845$ ,  $b = 17.998$ ,  $c = 10.143$  Å (at 173K),  $D_m = 2.10$ ,  $Z = 8$ . Mo radiation,  $R = 0.065$  for 1568 reflexions.

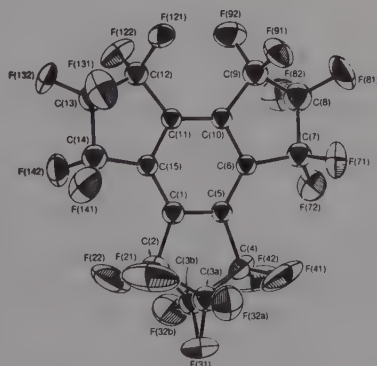


Fig. 1. A view of the  $C_{15}F_{18}$  molecule; C(3) and F(32) are disordered with the ratio of occupancies for a:b of 0.3955:0.6045.

In the molecule (Fig. 1) the perfluorocyclopentyl rings are non-planar and have a partially opened envelope conformation. The geometry and planarity of the six-membered carbon core of the molecule are consistent with complete electron delocalization in the ring, the mean C-C distance and mean C-C-C angle being  $1.390(14)$  Å and  $119.9(5)^\circ$  respectively. The geometry of the disordered perfluoro-

cyclopentyl ring is, not surprisingly, not totally consistent with those of the two ordered rings. There are no short intermolecular interactions.

# CYCLOBUTA[1]PHENANTHRENE-1,2-DIONE



N.P. HACKER, J.F.W. MCOMIE, J. MEUNIER-PIRET and M. VAN MEERSSCHE, 1982. J. Chem. Soc. Perkin I, 19-23.

Triclinic,  $\bar{P}1$ ,  $a = 9.762$ ,  $b = 9.129$ ,  $c = 7.049$  Å,  $\alpha = 107.71^\circ$ ,  $\beta = 97.97^\circ$ ,  $\gamma = 64.45^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.047$  for 1370 reflexions.

The bond lengths in the cyclobutene ring (Fig. 1) are C(3)-C(4) 1.580, C(3)-C(6) (mean) 1.489, and C(5)-C(6) 1.362 Å. These distances and the C(3)-O(1) distance of 1.194 Å (mean) agree well with expected values. The bond angles around the cyclobutene ring are C(3)-C(4)-C(5) (mean) 85.8, C(4)-C(5)-C(6) (mean) 94.2, C(4)-C(3)-O(1) (mean) 137.1 and C(5)-C(6)-C(7) (mean) 123.5°.

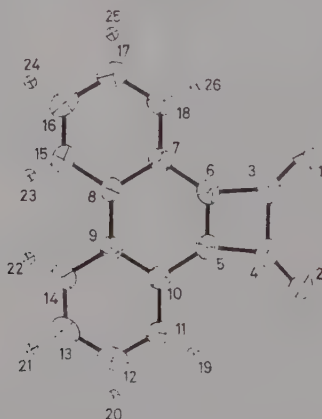


Fig. 1.  $\text{C}_{16}\text{H}_8\text{O}_2$ : the molecular structure.

# BENZ[a]ANTHRACENE-7,12-DIONE



R. KURODA, S. NEIDLE and A. SUBBIAH, 1982. Acta Cryst., B38, 1674-1676.

Monoclinic,  $C2/c$ ,  $a = 10.918$ ,  $b = 11.369$ ,  $c = 19.850$  Å,  $\beta = 97.22^\circ$ ,  $D_m = 1.41$ ,  $Z = 8$ . Cu radiation,  $R = 0.045$  for 1039 reflexions.

In this almost planar molecule (Fig. 1) rings A and D are twisted at  $7^\circ$  to each other. Ring C is significantly non-delocalised with C(5)-C(6) 1.348(4) Å; C(3)-C(4) is 1.328(5) Å. The C-C bonds adjacent to C=O are longer than normal (mean 1.485 Å) while C(7a)-C(11a) and C(6a)-C(12a) are shorter at 1.383(3) and 1.386(3) Å. Molecules are linked by van der Waals forces.

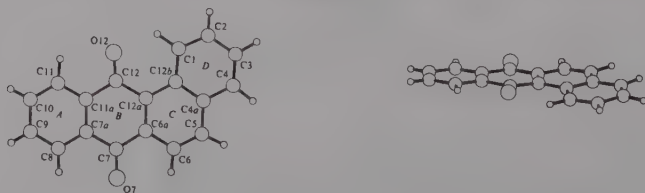


Fig. 1. Two views of the benz[a]anthracene-7,12-dione molecule.





The crystal structure (Fig. 1) contains discrete molecules with approximate twofold symmetry which are separated by normal van der Waals distances. Bond lengths are generally normal except for the distances C(6)-C(19) and C(9)-C(16), both 1.567(2) Å, which are much longer than expected for C(sp<sup>3</sup>)-C(sp<sup>3</sup>) bonds; this could result from strain in the molecule.

[5]HELICENE



R. KURODA, 1982. J. Chem. Soc. Perkin II, 789-794.

I. Monoclinic, P2<sub>1</sub>/c,  $a = 5.8150$ ,  $b = 14.178$ ,  $c = 17.498$  Å,  $\beta = 94.41^\circ$ ,  $D_m = 1.28$ ,  $Z = 4$ . Cu radiation,  $R = 0.060$  for 2537 reflexions.

II. Monoclinic, A2/a,  $a = 26.013$ ,  $b = 8.898$ ,  $c = 14.494$  Å,  $\beta = 103.94^\circ$ ,  $D_m = 1.27$ ,  $Z = 12$ . Cu radiation,  $R = 0.068$  for 2124 reflexions.

There are two crystallographically independent molecules (A,B) in II; A lies on a general position and B on a site of twofold symmetry. The molecules of I and II on general positions have approximate C<sub>2</sub> symmetry. The molecular structures are generally similar and dimensions follow the classification of medium, inner, external and radial bonds for helicenes. Molecule B in II shows slightly different dimensions from the other two molecules but these may not be significant on account of the high e.s.d.'s, particularly in the inner part of the helix. The closest intramolecular contacts in each are between atoms C(1) and C(14) (2.921, 2.954, 2.925 Å). Deviations of up to 30° from the ideal values of 0 and 180° for the torsion angles for regular aromatic hydrocarbons are observed in the inner parts of the helices. In each helix the terminal rings are planar and the central rings markedly puckered. Dihedral angles between neighbouring rings are 12-13°. In the crystal, molecules of I (Fig. 1) are aligned such that their C<sub>2</sub> axes are parallel to the c-axis throughout the structure. There are homochiral layers parallel to the ab plane with alternate layers of R and S molecules along c. The closest intermolecular contacts are between molecules related by a-axis translations ( $a = 5.815$  Å). The mean planes of molecules related by a 2<sub>1</sub> axis form a dihedral angle of 82.7° and the usual herring-bone pattern is observed in the ab plane. In II there are layers of solely A molecules and of B molecules parallel to the ab plane. In the layers of A molecules, molecules of opposite chirality are related by inversion centers along the c-axis and the homochiral molecules by a b-axis translation. Layers of B molecules are composed of homochiral molecules related by a translation and 2<sub>1</sub> axis along b. The layers of R molecules and those of S alternate with the layer of A molecules between them. The crystal packing is compared with that of other helicenes.



Fig. 1. C<sub>22</sub>H<sub>14</sub>: projections of the packing, I, along a (left), and II, along b, (right).



I. IKEMOTO, Y. IYETIKA, K. YAKUSHI, H. KURODA and K. PALEWSKA, 1982. Acta Cryst., B38, 988-991.

Orthorhombic,  $Pbca$ ,  $a = 18.338$ ,  $b = 23.460$ ,  $c = 8.330$  Å,  $D_m = 1.18$ ,  $Z = 8$ . Mo radiation,  $R = 0.057$  for 2352 reflexions.

The molecule (Fig. 1) can be regarded as a composite in which two 1,2-substituted naphthalene units are linked to hydrogenated rings. The central hydrogenated rings (C and D) have, respectively, the half-boat and half-chair conformations, with average C-C single-bond length of 1.522 Å. The C-C bond lengths in rings A, B, E and F average 1.397 Å. The dihedral angle between two naphthalene rings is 68.1°. No intermolecular distance is shorter than a van der Waals contact.

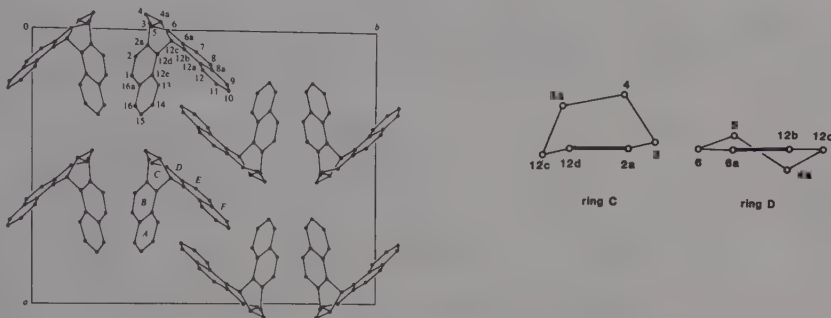


Fig. 1.  $C_{26}H_{22}$ : projection of the crystal structure along the  $c$  axis (left) and the conformation of the central hydrogenated rings (right).

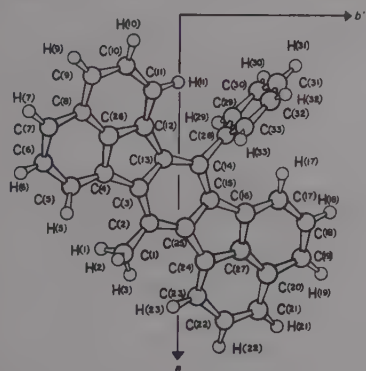
#### 14-METHYL-7-PHENYLACENAPHTHO[1,2-k]FLUORANTHENE

$C_{33}H_{20}$

S. SETH and S. CHAKRABORTY, 1982. *Acta Cryst.*, **B38**, 1380-1383.

Triclinic,  $P\bar{1}$ ,  $a = 12.184$ ,  $b = 12.507$ ,  $c = 8.542$  Å,  $\alpha = 97.64$ ,  $\beta = 104.45$ ,  $\gamma = 116.19^\circ$ ,  $D_m = 1.26$ ,  $Z = 2$ . Mo radiation,  $R = 0.051$  for 2467 reflexions.

In the molecule (Fig. 1) the fluoranthene moiety is slightly non-planar; the phenyl ring is almost perpendicular to it and is essentially planar with normal dimensions. Bond lengths and angles are in reasonably good agreement with those in other nonalternant hydrocarbons.



C(1)-C(2)	1.504 (3)	C(2)-C(3)	1.398 (3)
C(2)-C(25)	1.397 (3)	C(3)-C(4)	1.479 (3)
C(3)-C(13)	1.424 (3)	C(4)-C(5)	1.382 (3)
C(4)-C(26)	1.415 (3)	C(5)-C(6)	1.420 (4)
C(6)-C(7)	1.363 (4)	C(7)-C(8)	1.418 (3)
C(8)-C(9)	1.417 (4)	C(8)-C(26)	1.400 (3)
C(9)-C(10)	1.366 (4)	C(10)-C(11)	1.421 (4)
C(11)-C(12)	1.374 (3)	C(12)-C(13)	1.474 (3)
C(12)-C(26)	1.414 (3)	C(13)-C(14)	1.397 (3)
C(14)-C(15)	1.393 (3)	C(14)-C(28)	1.490 (3)
C(15)-C(16)	1.480 (3)	C(15)-C(25)	1.422 (3)
C(16)-C(17)	1.377 (3)	C(16)-C(27)	1.413 (3)
C(17)-C(18)	1.418 (4)	C(18)-C(19)	1.375 (4)
C(19)-C(20)	1.416 (4)	C(20)-C(21)	1.421 (4)
C(20)-C(27)	1.395 (3)	C(21)-C(22)	1.361 (4)
C(22)-C(23)	1.417 (4)	C(23)-C(24)	1.378 (3)
C(24)-C(25)	1.486 (3)	C(24)-C(27)	1.414 (3)
C(28)-C(29)	1.385 (3)	C(28)-C(33)	1.383 (3)
C(29)-C(30)	1.388 (3)	C(30)-C(31)	1.373 (4)
C(31)-C(32)	1.377 (4)	C(32)-C(33)	1.396 (4)

Fig. 1.  $C_{33}H_{20}$ : a perspective view of the molecule and bond lengths.

(9S,P)-(+)-7,9-DI-*t*-BUTYL-9H-NAPHTHO[1',2':3,4]CYCLOPENTA[1,2-*c*]PHENANTHRENE  
C<sub>33</sub>H<sub>32</sub>

H. NAKAI, M. SHIRO, S. IMAJO, H. KURITANI and K. SHINGU, 1982. *Acta Cryst.*, B38, 2715-2718.

Orthorhombic, P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 11.148, *b* = 22.163, *c* = 9.998 Å, *Z* = 4. Cu radiation, *R* = 0.056 for 2161 reflexions.

The molecule (Fig. 1) has a spiral structure, i.e. a rel-(9S,P) configuration. Distortion from planarity is significant for all the benzene rings. The cyclopentadiene ring is deformed into an envelope form. The consecutive ring planes are considerably bent. The molecule is more stable in the (9S,P) than in the (9S,M) configuration.

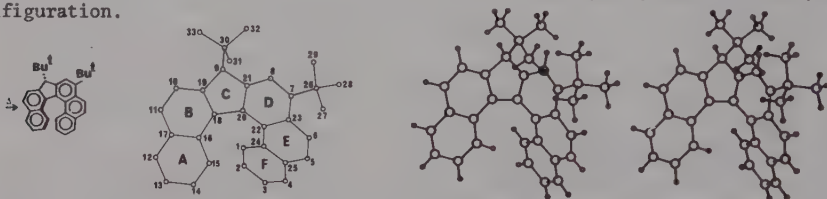


Fig. 1. C<sub>33</sub>H<sub>32</sub>: perspective and stereoscopic views of the molecule.

ANTHRA[2,1,9,8-hijk]BENZO[de]NAPHTHO[2,1,8,7-stuv]PENTACENE  
C<sub>38</sub>H<sub>18</sub>



S. FUJISAWA, I. OONISHI, J. AOKI, Y. OHASHI and Y. SASADA, 1982. *Bull. Chem. Soc. Jpn.*, 55, 3424-3428.

R.E. MARSH and F.H. HERBSTSTEIN, 1988. *Acta Cryst.*, B44, 77-88.

Monoclinic, *Pa*, *a* = 31.276, *b* = 3.7818, *c* = 21.663 Å, β = 124.22°, *Z* = 4. Cu radiation, *R* = 0.073 for 2619 reflexions.

Monoclinic, P2<sub>1</sub>/*a*, *a* = 31.276, *b* = 3.7818, *c* = 21.663 Å, β = 124.22°, *Z* = 4, Cu radiation, *R* = 0.069 for 2619 reflexions.

Fujisawa et al., report the structure in space group *Pa* with two independent molecules. Marsh and Herbstein show that it should be in P2<sub>1</sub>/*a* with one molecule per asymmetric unit. The molecule is distorted from planarity because of repulsions between the crowded hydrogen atoms.

6-OXO-BICYCLO[2.2.1]HEPTANE-endo-2-CARBOXYLIC ACID  
C<sub>8</sub>H<sub>10</sub>O<sub>3</sub>

D.J. CHADWICK, S.N. WHITTLETON and R.W.H. SMALL, 1982. *J. Chem. Soc. Perkin II*, 669-675.

Monoclinic, P2<sub>1</sub>/*c*, *a* = 6.703, *b* = 11.836, *c* = 10.918 Å, β = 121.6°, *Z* = 4. Mo radiation, *R* = 0.063 for 1298 reflexions.

The endocyclic angle at the carbonyl function C(1)-C(2)-C(3) (Fig. 1) is 105.4° and the exocyclic angles O(1)-C(2)-C(1) and O(1)-C(2)-C(3) are 128.3 and 126.3° respectively. The C(2)-O(1) bond length is 1.192 Å. The carbonyl group is slightly pyramidal, C(2) being displaced by 0.015 Å out of the plane of O(1), C(1), C(3) towards the carboxyl group. The crystal structure contains only the γ-keto acid with none of the possible hydroxy lactone isomer present. The acid is present as hydrogen bonded dimers in the crystal.

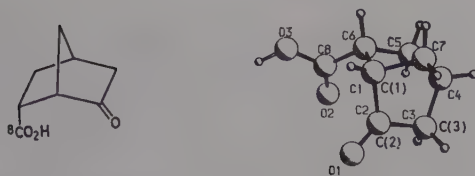


Fig. 1. Molecular skeleton and structure of  $C_8H_{10}O_3$ .

2-endo,6-endo-BIS(METHYLTHIO)-3-exo,5-exo-DICHLOROBICYCLO[2.2.1]HEPTANE  
 $C_9H_{14}Cl_2S_2$

R.S. GLASS, B.R. COLEMAN, U.D.G. PRABHU, W.N. SETZER and G.S. WILSON, 1982. J. Org. Chem., **47**, 2761-2764.

Orthorhombic,  $Pnma$ ,  $a = 9.483$ ,  $b = 17.879$ ,  $c = 7.137$  Å,  $D_m = 1.404$ ,  $Z = 4$ . Mo radiation,  $R = 0.039$  for 710 reflexions.

The molecule (Fig. 1) has crystallographic mirror symmetry, and the S...S separation is short (3.179(1) Å).

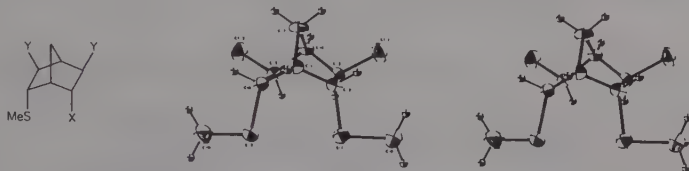


Fig. 1. The  $C_9H_{14}Cl_2S_2$  molecule ( $X = SMe$ ,  $Y = Cl$ ).

2,3-DIHYDROXY-1-METHYLBICYCLO[3.2.1]OCTAN-8-ONE HYDRATE  
 $C_9H_{14}O_3 \cdot H_2O$  (I)

9,11-METHANO-10-OXAPENTACYCLO[5.3.3.0.0<sup>2,4</sup>]TRIDECAN-8-ONE  
 $C_{13}H_{16}O_2$  (II)

A. GILLON, D. OVADIA, M. KAPON and S. BIEN, 1982. Tetrahedron, **38**, 1477-1484.

I. Monoclinic,  $P2_1/c$ ,  $a = 12.040$ ,  $b = 6.727$ ,  $c = 12.522$  Å,  $\beta = 100.99^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.077$  for 965 reflexions.

II. Orthorhombic,  $P2_12_12_1$ ,  $a = 15.897$ ,  $b = 10.104$ ,  $c = 6.429$  Å,  $Z = 4$ . Mo radiation,  $R = 0.063$  for 801 reflexions.

For I, the six-membered ring of the bicyclic skeleton (Fig. 1) has a distorted chair conformation with unusual folding along the C(1)-C(5) vector and flattening along the C(2)-C(4) vector. However the ring still maintains a point group symmetry  $C_s$  where the mirror plane bisects atoms C(3) and C(8). In II, the six-membered ring as a part of a bicyclo[4.1.0] heptane system, has the expected "half-chair" conformation. The torsion angle C(3)-C(4)-C(6)-C(7),  $-2.3^\circ$  and the bond angles C(3)-C(4)-C(6),  $121.1^\circ$ , C(4)-C(6)-C(7),  $119.4^\circ$ , provide a geometry comparable with that of a group where a double bond replaces the cyclopropane ring at C(4)-C(6). The apex atom C(5) of the cyclopropane ring is disposed on the same side of the six-membered ring with respect to the heterocyclic oxygen O(2).

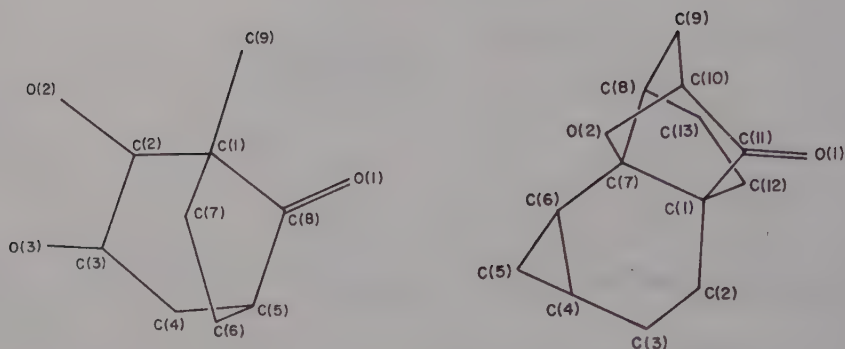


Fig. 1. The  $C_9H_{14}O_3$  (I) (left) and  $C_{13}H_{16}O_2$  (II) (right) structures.

exo-2,exo-6-DIHYDROXYBICYCLO[3.3.1]NONANE  
 $C_9H_{16}O_2$  (I)

exo-2,exo-6-DIHYDROXY-2,6-DIETHYLBICYCLO[3.3.1]NONANE  
 $C_{13}H_{24}O_2$  (II)

R. BISHOP, S. CHOUDHURY and I. DANCE, 1982. J. Chem. Soc. Perkin II, 1159-1168.

I. Monoclinic,  $P2_1/c$ ,  $a = 12.970$ ,  $b = 13.591$ ,  $c = 11.150$  Å,  $\beta = 118.68^\circ$ ,  $D_m = 1.19$ ,  $Z = 8$ . Cu radiation,  $R = 0.046$  for 3049 reflexions.

II. Orthorhombic,  $Fdd2$ ,  $a = 12.756$ ,  $b = 16.661$ ,  $c = 23.266$  Å,  $D_m = 1.15$ ,  $Z = 16$ . Cu radiation,  $R = 0.050$  for 1083 reflexions.

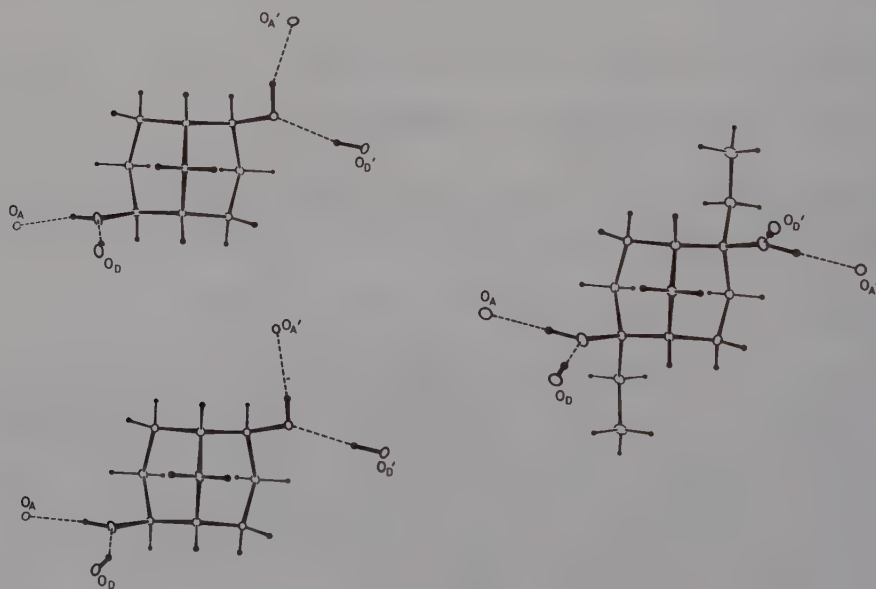


Fig. 1. Molecular structures and hydrogen bonding patterns for  $C_9H_{16}O_2$  (I) (left) and  $C_{13}H_{24}O_2$  (II) (right).



There are two molecules in the asymmetric unit of I. The structures of I and II contain the maximum number of possible intermolecular hydrogen bonds, but with quite different networks which differ in the  $O_D \dots O \dots O_A$  and  $O_D' \dots O' \dots O_A'$  sector angles. In I they are distributed closely around the tetrahedral value, and in II they are both small ( $81.6^\circ$ ,  $85.0^\circ$ ). In the crystal structure of I the molecules are stacked along the *b* direction with a pseudo-hexagonal array of stacks. The C(3)-O bond makes angles of  $54.1$  and  $31.3^\circ$  with the stacking axis. The hydrogen bonding network consists of infinite connections between hydroxyl groups in which inter and intra stack linkages alternate, extension occurring according to the *c*-glide operation. In II the hydrogen bonding connections are not infinite but occur in four membered cycles. The molecules are stacked along  $2_1$  screw axes parallel to *c*. The hydrogen bonds protrude from those stacks approximately normal to the  $2_1$  axes, making links between four stacks around a twofold axis.

#### 6-NITROBENZONORBORNEN-2-ONE

$C_{11}H_9NO_3$

T. REITZ, G.L. GRUNEWALD, C.H. PARK and B.K. LEE, 1982. *Cryst. Struct. Comm.*, **11**, 1659-1662.

Triclinic,  $P\bar{1}$ ,  $a = 6.251$ ,  $b = 9.208$ ,  $c = 9.389$  Å,  $\alpha = 95.60^\circ$ ,  $\beta = 108.37^\circ$ ,  $\gamma = 109.58^\circ$ ,  $D_m = 1.40$ ,  $Z = 2$ . Mo radiation,  $R = 0.046$  for 1839 reflexions.

The analysis establishes that the nitro group is at C6 (Fig. 1). The norbornenone nucleus is strained, with C1-C9  $95.1^\circ$ .

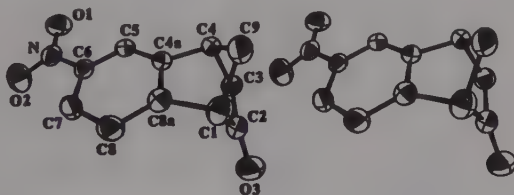


Fig. 1. The 6-nitrobenzonorbornen-2-one molecule; a stereoview.

#### PENTAPRISMANE CARBOXYLIC ACID

$C_{11}H_{10}O_2$

P. ENGEL, P.E. EATON and B.K.R. SHANKAR, 1982. *Z. Krist.*, **159**, 239-246.

Monoclinic,  $P2_1/c$ ,  $a = 6.421$ ,  $b = 10.766$ ,  $c = 11.871$  Å,  $\beta = 104.8^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.049$  for 1397 reflexions.

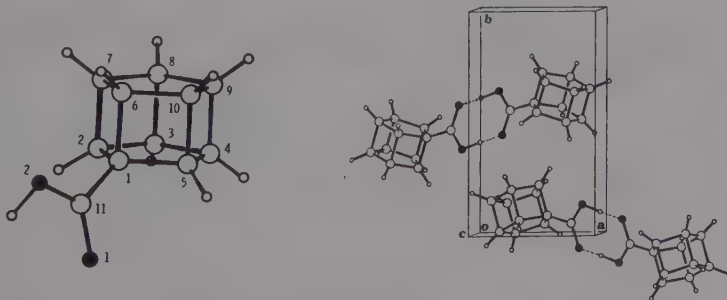


Fig. 1. The  $C_{11}H_{10}O_2$  molecule and packing (the O-H...O hydrogen bond has length 2.64 Å).



Pentaprismane (Fig. 1) shows almost perfect  $D_{5h}$  symmetry; distortions due to the carboxylic acid group remain small. Two hydrogen bonds between the carboxylic acid groups bind two molecules in the crystal structure. These paired molecules are bonded by van der Waals forces only. The mean C-C within the five-membered rings is 1.548(8) Å and the C-C bonds parallel to the prism axis have mean value 1.565(4) Å.

4-BROMO-5-OXO-TETRACYCLO[4.3.0.0<sup>2,4</sup>.0<sup>3,8</sup>]NONAN-7-(exo)CARBOXYLIC ACID METHYL ESTER  
 $C_{11}H_{11}BrO_3$

J.H. NOORDIK, E.V.D. LOOP, A.J.H. KLUNDER and G.J.A. ARIAANS; 1982. *Cryst. Struct. Comm.*, **11**, 1343-1346.

Monoclinic,  $P2_1/n$ ,  $a = 7.470$ ,  $b = 6.342$ ,  $c = 21.583$  Å,  $\beta = 90.26^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.052$  for 1015 reflexions.

The X-ray analysis established the structure of this strained hydrocarbon to be as shown in Fig. 1.

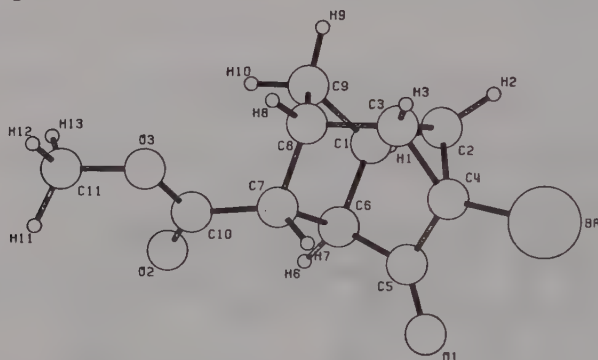


Fig. 1. A view of  $C_{11}H_{11}BrO_3$ .

1,5-BIS(ACETOXYMETHYL)TRICYCLO[2.1.0.0<sup>2,5</sup>]PENTAN-3-ONE  
 $C_{11}H_{12}O_5$

P. DOWD, P. GARNER, R. SCHAPPERT, H. IRNGARTINGER and A. GOLDMAN, 1982. *J. Org. Chem.*, **47**, 4240-4246.

Monoclinic,  $P2_1/c$ ,  $a = 6.5497$ ,  $b = 12.654$ ,  $c = 13.887$  Å,  $\beta = 101.902^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.05$  for 1625 reflexions.

The molecule and dimensions are shown in Fig. 1. Bond length C(2)-C(3) (1.416(2) Å) is very short and the bond has pronounced p-character. The dihedral angle between the two cyclopropane rings is  $95.7^\circ$ .

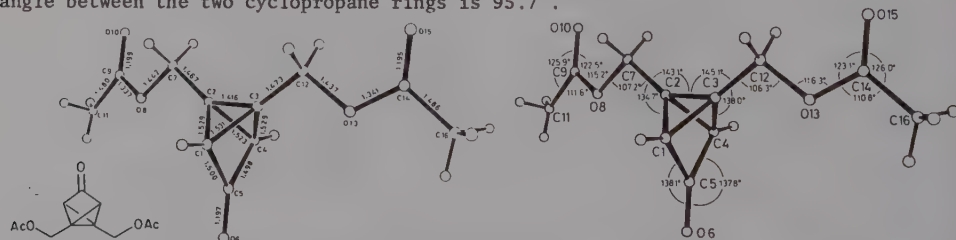


Fig. 1. The structure of  $C_{11}H_{12}O_5$  with bond lengths ( $\sigma$  0.002-0.006 Å) and angles ( $\sigma$  0.1-0.3°).

5-endo-(BROMOMETHYL)-1,2,4-TRICHLORO-3,7,7-TRIMETHOXYNORBORN-2-ENE  
 $C_{11}H_{14}BrCl_3O_3$

D. VAN HEMELRIJK, A.T.H. LENSTRA and H.J. GEISE, 1982. Acta Cryst., B38, 2760-2763.

Monoclinic,  $P2_1/n$ ,  $a = 7.988$ ,  $b = 15.672$ ,  $c = 11.949$  Å,  $\beta = 103.90^\circ$ ,  $Z = 4$ . Mo radiation,  $R_w = 0.071$  for 1478 reflexions.

The geometry of the title compound (Fig. 1) is compared with a number of closely related molecules. Anomeric interaction governs the shape of the  $CH_3-O-C(7)-O-CH_3$  moiety. The bicyclo[2.2.1]-hept-2-ene skeleton is hardly distorted, despite heavy substitution. Rather the geometry of substituents is influenced by the skeleton causing the inequivalence of many seemingly equivalent valence angles, e.g.  $C(4)-C(7)-O(8)$  and  $C(1)-C(7)-O(9)$  are about  $10^\circ$  larger than  $C(4)-C(7)-O(9)$  and  $C(1)-C(7)-O(8)$ .

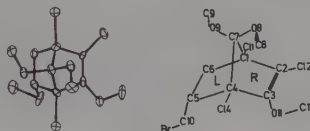


Fig. 1.  $C_{11}H_{14}BrCl_3O_3$ : conformation of the molecule and numbering of the atoms.

2-NITRO-3-METHOXYCARBONYLMETHYLBICYCLO[2.2.2]OCTAN-5-ONE  
 $C_{11}H_{15}NO_5$

A.I. AKHMEDOV, A.I. YANOVSKY and Yu.T. STRUCHKOV, 1982. Cryst. Struct. Comm., 11, 185-189.

Monoclinic,  $P2_1/c$ ,  $a = 10.996$ ,  $b = 10.814$ ,  $c = 10.099$  Å,  $\beta = 105.24^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.054$  for 1508 reflexions.

The analysis establishes the structure of this reaction product to be as shown in Fig. 1.

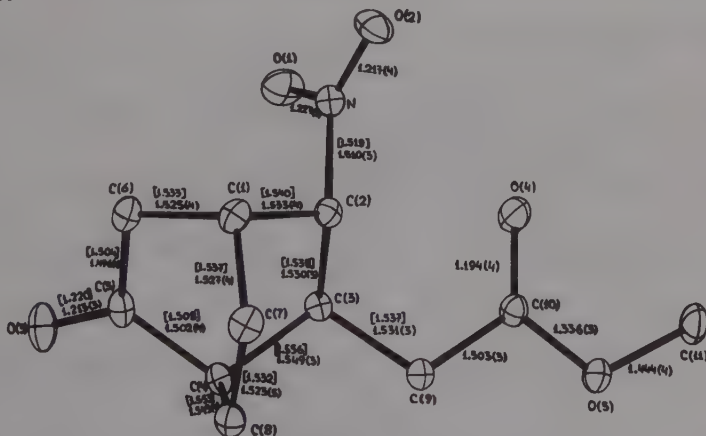


Fig. 1. The structure of  $C_{11}H_{15}NO_5$  with bond lengths (corrected for libration in square brackets).

## 3,3,6,6-TETRAMETHYLBICYCLO[2.2.1]HEPTANE-2,5-DIONE

 $C_{11}H_{16}O_2$  (I)

## 6,6-DIBROMO-1,3,3-TRIMETHYLBICYCLO[2.2.2]OCTANE-2,5-DIONE

 $C_{11}H_{14}Br_2O_2$  (II)

R. FAGGIANI, C.J.L. LOCK, N.H. WERSTIUK and S. YEROUSHALMI, 1982. *J. Cryst. Spectrosc. Res.*, **12**, 351-362.

I. Monoclinic,  $C2/c$ ,  $a = 9.071$ ,  $b = 9.748$ ,  $c = 12.322$  Å,  $\beta = 110.15^\circ$ ,  $D_m = 1.14$ ,  $Z = 4$ . Mo radiation,  $R = 0.065$  for 570 reflexions.

II. Orthorhombic,  $Pcab$ ,  $a = 11.206$ ,  $b = 18.818$ ,  $c = 11.695$  Å,  $D_m = 1.76$ ,  $Z = 8$ . Mo radiation,  $R = 0.070$  for 1210 reflexions.

Molecule I has twofold crystallographic symmetry (Fig. 1). Molecule II is obtained from I by rearrangement. The opening up of the internal ring angle of the ketone in I and the consequent decrease of the adjacent internal ring angle brings the ketone carbon atoms nearer to the hydrogen atoms of the bridging methylene group.

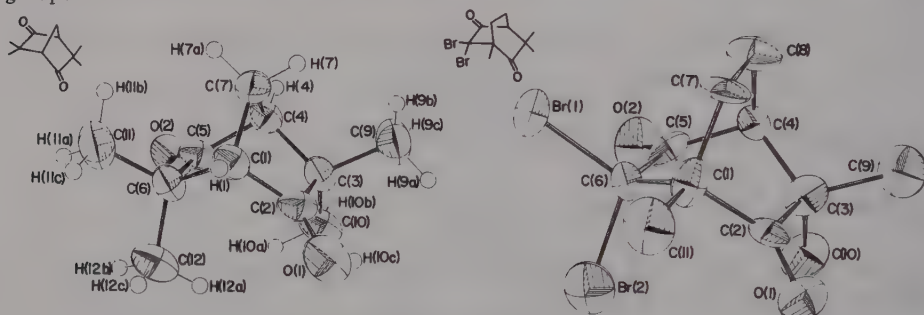


Fig. 1. The  $C_{11}H_{16}O_2$  and  $C_{11}H_{14}Br_2O_2$  molecules.

4-exo-iodo-anti-6-perchloryloxy-9,10-cis-dimethyltetracyclo[5.3.0.0<sup>2,5</sup>.0<sup>3,8</sup>]decane $C_{12}H_{15}ClIO_4$ 

K.A. POTEKHIN, V.G. RAU, Yu.T. STRUCHKOV, V.V. ZHDANKIN, A.S. KOZ'MIN and N.S. ZEFIROV, 1982. *Cryst. Struct. Comm.*, **11**, 211-214.

Triclinic,  $P\bar{1}$ ,  $a = 6.573$ ,  $b = 10.379$ ,  $c = 10.672$  Å,  $\alpha = 104.77^\circ$ ,  $\beta = 102.46^\circ$ ,  $\gamma = 96.09^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.043$  for 3588 reflexions.

The structure shown in Fig. 1 is established, with the perchloryloxy group anti with respect to the iodine atom.

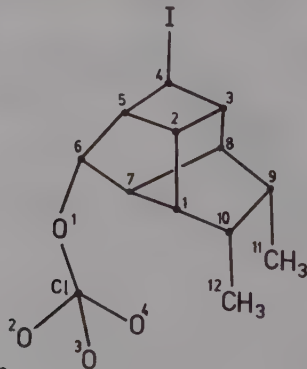
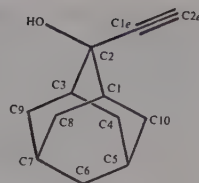


Fig. 1. The  $C_{12}H_{15}ClIO_4$  molecule.

## 2-ETHYNYL-2-ADAMANTANOL

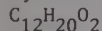


S.Y. LIN, Y. OKAYA, D.M. CHIOU and W.J. LE NOBLE, 1982. *Acta Cryst.*, **B38**, 1669-1671.

Monoclinic,  $P2_1/c$ ,  $a = 6.820$ ,  $b = 22.644$ ,  $c = 12.708$  Å,  $\beta = 94.5^\circ$ ,  $D_m = 1.19$ ,  $Z = 8$ . Cu radiation,  $R = 0.050$  for 2200 reflexions.

The two independent molecules have very similar, normal geometries. They have approximate mirror planes through C2, C5, C6, C7, with the ethynyl group and hydroxyl O lying within 0.08 Å of this plane. The molecules are linked by O-H...O (2.839 Å) hydrogen bonds and one molecule has an O-H... $\pi$  intramolecular hydrogen bond; the C(2)-O bond lengths 1.452(2) and 1.428(2) Å may reflect this difference in the hydrogen bonding scheme. The C(1e)-C(2e) bond lengths are 1.180(3) and 1.170(3) Å.

## 2,3-DIMETHYL-4-VINYLBICYCLO[3.2.1]OCTAN-2,3-DIOL



N. LAMB, T. MONEY, S.J. RETTIG and J. TROTTER, 1982. *Canad. J. Chem.*, **60**, 1055-1061.

Orthorhombic,  $C222_1$ ,  $a = 9.571$ ,  $b = 12.416$ ,  $c = 18.860$  Å,  $D_m = 1.15$ ,  $Z = 8$ . Mo radiation,  $R = 0.035$  for 930 reflexions.

The X-ray analysis identified this reaction product to be as shown in Fig. 1. The five-, six-, and seven-membered rings comprising the bicyclo[3.2.1]octane system have envelope, distorted chair and distorted boat conformations respectively. There is an intramolecular O-H...O hydrogen bond (O1...O2 2.502(3) Å), and each molecule is linked to two others by O-H...O hydrogen bonds (O...O 2.805(4), 2.803(4) Å).

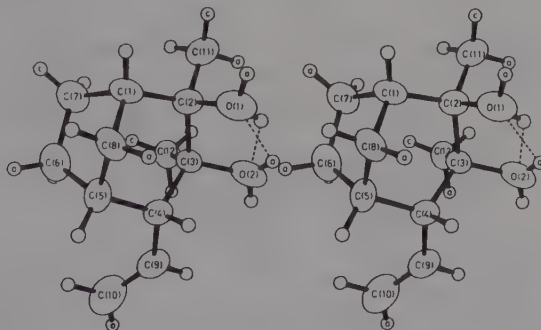
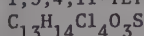


Fig. 1. A stereoview of  $\text{C}_{12}\text{H}_{20}\text{O}_2$ . The hydroxyl hydrogens are disordered over two sites.

1,3,4,11-TETRACHLORO-2-METHYLSULFONATOMETHYLTETRACYCLO[5.3.1.0<sup>2,6</sup>.0<sup>5,9</sup>]UNDEC-3-ENE

K.A. MEAD, K. MACKENZIE and P. WOODWARD, 1982. *J. Chem. Soc. Perkin II*, 571-578.

Monoclinic,  $P2_1/n$ ,  $a = 7.693$ ,  $b = 17.007$ ,  $c = 11.894$  Å,  $\beta = 98.78^\circ$ ,  $D_m = 1.64$ ,  $Z = 4$ . Mo radiation,  $R = 0.042$  for 3396 reflexions.

The carbon skeleton (Fig. 1) has three fused five-membered rings with C(6) in common. The angles around C(6) are approximately tetrahedral, C(7)-C(6)-C(5) 106.2, C(2)-C(6)-C(7) 108.0 and C(5)-C(6)-C(2) 100.9°. All three rings have envelope conformations. The dichloro olefin fragment C(2),C(3),Cl(3),C(4),Cl(4),C(5) is planar. The Cl(11)-C(11)-C(1)-Cl(1) torsion angle is 49.0 while the fragments C(2)-C(6)-C(7)-C(11) (torsion angle -0.3) and C(5)-C(6)-C(7)-C(8) (torsion angle -4.0°) are almost planar.

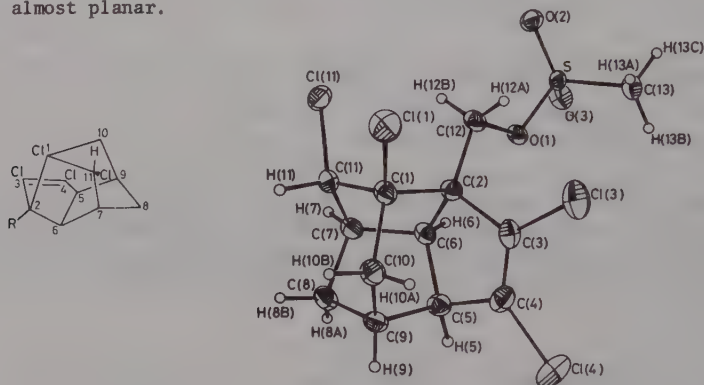


Fig. 1. Molecular skeleton (R = CH<sub>2</sub>OSO<sub>2</sub>Me) and structure of C<sub>13</sub>H<sub>14</sub>Cl<sub>4</sub>O<sub>3</sub>S.

11-OXO-5,6,7,8,9,10-HEXAHYDRO-6,9-METHANOBENZOCYCLOOCTENE  
C<sub>13</sub>H<sub>14</sub>O

P.C. BÉLANGER, R.N. YOUNG, J. SCHEIGETZ, C. DUFRESNE, J.P. SPRINGER, 1982. *J. Org. Chem.*, **47**, 4329-4334.

Monoclinic, P2<sub>1</sub>/c, a = 6.773, b = 12.767, c = 23.064 Å, β = 93.28°, Z = 8. R = 0.050 for 2098 reflexions.

There are two independent molecules in the asymmetric unit related by a pseudo inversion centre. The cyclooctene ring has a boat conformation (Fig. 1).

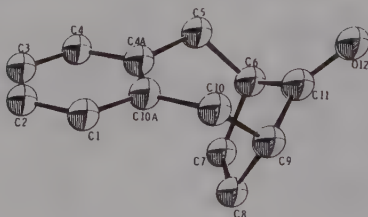


Fig. 1. A view of one of the C<sub>13</sub>H<sub>14</sub>O molecules.

11-exo-AMMONIO-8-CHLOROBENZO[b]BICYCLO[3.3.1]NONA-2,6a(10a)-DIENE CHLORIDE

C<sub>13</sub>H<sub>15</sub>Cl<sub>2</sub>N<sup>+</sup>

C<sub>13</sub>H<sub>15</sub>ClN<sup>+</sup>, Cl<sup>-</sup>

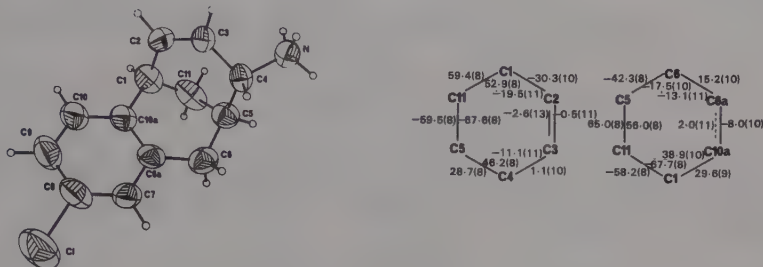
P.G. JONES and G.M. SHELDRICK, 1982. *Acta Cryst.*, **B38**, 3105-3107.

Orthorhombic, Pbca, a = 9.376, b = 16.353, c = 35.111 Å, Z = 16. Cu radiation, R = 0.071 for 1982 reflexions.

The two independent cations in the asymmetric unit have markedly different conformations (Fig. 1). The C(1)-C(2)-C(3)-C(4)-C(5)-C(11) rings show sofa and half-chair conformations for molecule 1 and 2 respectively. The C(6)-C(6a)-C(10a)-



C(1)-C(11)-C(5) rings are not close to ideal conformations, for molecule 1 being between half-chair ( $7^\circ$ ) and sofa ( $9^\circ$ ), for molecule 2 between sofa ( $9^\circ$ ) and 1,3-diplanar ( $9^\circ$ ) (mean absolute deviations in parentheses).





6SR)-PERCHLORYLOXY-9(RS)-HYDROXY-3,4-DIMETHOXYCARBONYLTETRACYCLO[6.1.1.0<sup>2,7</sup>.0<sup>5,10</sup>]-  
DEC-3-ENE  
C<sub>14</sub>H<sub>15</sub>ClO<sub>9</sub>

D.S. YUFIT, Yu.T. STRUCHKOV, N.M. YUR'EVA, A.S. KOZ'MIN and N.S. ZEFIROV, 1982.  
Cryst. Struct. Comm., 11, 1903-1908.

Monoclinic, C2/c, a = 19.668, b = 8.689, c = 18.382 Å, β = 108.10°, Z = 8. Mo  
radiation, R = 0.041 for 2980 reflexions (at -120°C).

The analysis shows that the molecule (Fig. 1) has a covalently bonded perchlorate  
with an endo-configuration, and a rearranged structure with a cross-bonded skeleton.  
Molecules are linked into dimers around twofold symmetry axes by weak O-H...O  
hydrogen bonds (O...O 2.699(3) Å).

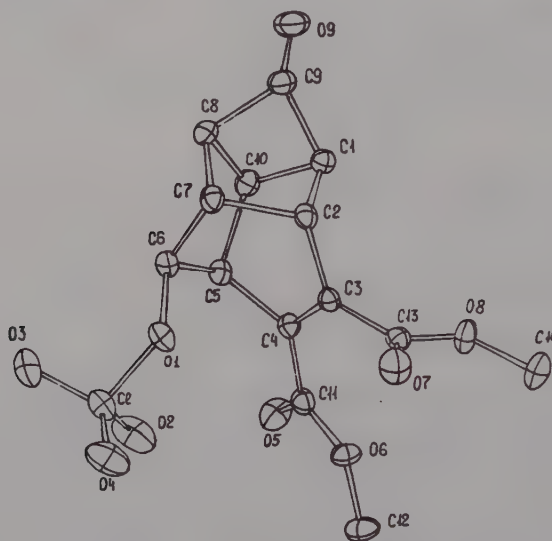


Fig. 1. A view of a molecule of C<sub>14</sub>H<sub>15</sub>ClO<sub>9</sub>.

9(RS)-IODO-6-(SR)-PERCHLORYLOXY-3(RS),4(RS)-DIMETHOXYCARBONYLTETRACYCLO[6.1.1.-  
0<sup>2,7</sup>.0<sup>5,10</sup>]DECANE  
C<sub>14</sub>H<sub>16</sub>ClIO<sub>8</sub>

T.F. RAU, V.G. RAU, K.A. POTEKHIN, Yu.T. STRUCHKOV, V.V. ZHDANKIN, A.S. KOZ'MIN,  
V.N. KIRIN and N.S. ZEFIROV, 1982. Cryst. Struct. Comm., 11, 207-210.

Monoclinic, P2<sub>1</sub>/c, a = 8.947, b = 10.587, c = 17.922 Å, β = 86.75°, Z = 4. Mo  
radiation, R = 0.047 for 2029 reflexions.

The X-ray analysis establishes that the compound studied had the structure shown  
in Fig. 1, with a covalently bonded perchloryloxy group endo to the basal cyclohexane  
moiety.

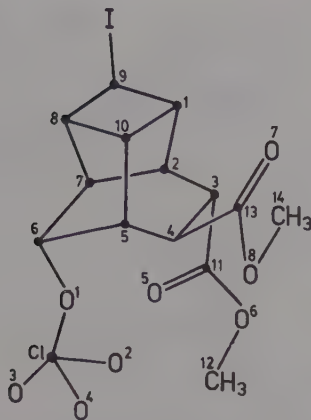
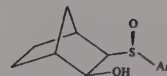


Fig. 1. The  $C_{14}H_{16}ClIO_8$  molecule.

exo-3-p-TOLYLSULPHINYLNORBORNAN-exo-2-OL  
 $C_{14}H_{18}O_2S$



A.L.J. BECKWITH, J.R. RODGERS and R.D. WAGNER, 1982. Aust. J. Chem., 35, 989-996.

Monoclinic,  $P2_1/n$ ,  $a = 10.863$ ,  $b = 15.182$ ,  $c = 15.826$  Å,  $\beta = 97.43^\circ$ ,  $D_m = 1.31$ ,  $Z = 8$ . Cu radiation,  $R = 0.0497$  for 2280 reflexions.

The analysis defined the structure to be that shown above.

(-)-4 $\alpha$ H-3 $\beta$ ,7 $\beta$ ,11-TRIMETHYLTRICYCLO[5.4.1<sup>1,4</sup>.0<sup>1,7</sup>]DODECA-10-EN-2 $\beta$ ,3 $\alpha$ -DIOL  
 $C_{15}H_{24}O_2$  (I)

(+)-4 $\alpha$ H-10 $\alpha$ ,11 $\alpha$ -EPOXY-3 $\alpha$ ,7 $\beta$ ,11 $\beta$ -TRIMETHYLTRICYCLO[5.4.1<sup>1,4</sup>.0<sup>1,7</sup>]DODECAN-2-ONE  
 $C_{15}H_{22}O_2$  (II)

(-)-4 $\alpha$ H-3 $\beta$ ,7 $\beta$ ,11-TRIMETHYLTRICYCLO[5.4.1<sup>1,4</sup>.0<sup>1,7</sup>]DODECA-10-EN-2,9-DIONE  
 $C_{15}H_{20}O_2$  (III)

S. INAYAMA, H. HORI, N. SHIMIZU, A.K. SINGH, T. KAWAMATA, T. SHIBATA and Y. IITAKA, 1982. Heterocycles, 19, 1813-1818.

I. Orthorhombic,  $P2_12_12_1$ ,  $a = 18.925$ ,  $b = 19.529$ ,  $c = 7.265$  Å,  $Z = 8$ . Cu radiation,  $R = 0.096$  for 1830 reflexions.

II. Orthorhombic,  $P2_12_12_1$ ,  $a = 8.798$ ,  $b = 18.929$ ,  $c = 7.793$  Å,  $Z = 4$ . Cu radiation,  $R = 0.096$  for 958 reflexions.

III. Monoclinic,  $P2_1$ ,  $a = 10.860$ ,  $b = 8.193$ ,  $c = 7.133$  Å,  $\beta = 92.49^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.087$  for 910 reflexions.

The structure of a tricyclic ketone has been unambiguously determined (Fig. 1) by means of the three crystal structure determinations listed above. The formula is (+)-4 $\alpha$ H-3 $\alpha$ ,7 $\beta$ ,11-trimethyltricyclo[5.4.1<sup>1,4</sup>.0<sup>1,7</sup>]dodecan-2-one.

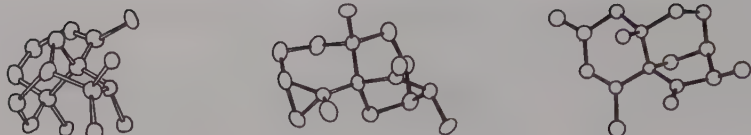


Fig. 1. The tricyclic ketones (I), (II) and (III) (left to right).

anti-1,6:8,13-BIS(DIFLUOROMETHANO) [14]ANNULENE

$C_{16}H_{10}F_4$

R. DESTRO and M. SIMONETTA, 1982. *Tetrahedron*, **38**, 1443-1446.

Orthorhombic,  $Cmc2_1$ ,  $a = 9.691$ ,  $b = 17.769$ ,  $c = 6.989$  Å,  $Z = 4$ . Mo radiation,  $R = 0.046$  for 1716 reflexions (at 200 K).

The molecule (Fig. 1) has crystallographic mirror symmetry. There is a systematic succession of long and short bond distances in the annulene perimeter.

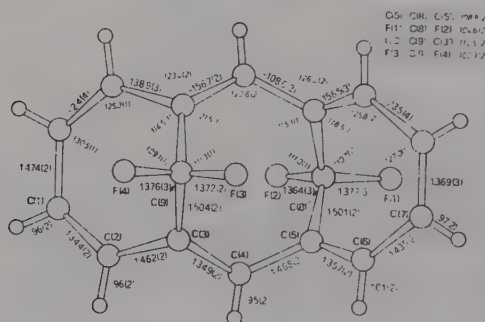


Fig. 1. The  $C_{16}H_{10}F_4$  molecule with bond lengths, angles and torsion angles.

endo,endo-4,5-DIACETOXY-exo-4-(METHOXYMETHYL)-exo-TRICYCLO[5.2.1.0<sup>2,6</sup>]DEC-8-EN-3-ONE

$C_{16}H_{20}O_6$

J.H. NOORDIK, V. PARTHASARATHI, A.J.H. KLUNDER and J.M. VERLAAK, 1982. *Cryst. Struct. Comm.*, **11**, 1339-1342.

Triclinic,  $P\bar{1}$ ,  $a = 11.465$ ,  $b = 10.573$ ,  $c = 6.965$  Å,  $\alpha = 105.98$ ,  $\beta = 104.82$ ,  $\gamma = 82.23^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.06$  for 1526 reflexions.

The analysis establishes the structure of this reaction product as shown in Fig. 1. The torsion angle O2-C2-C1-O1 is  $-5.3(4)^\circ$ .

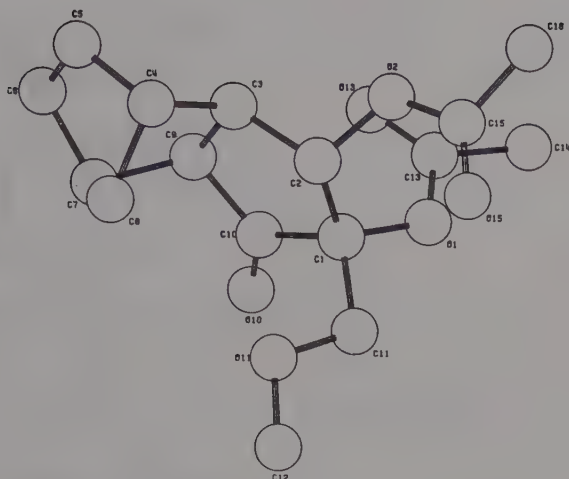


Fig. 1. A view of  $C_{16}H_{20}O_6$ .

10-ACETAMIDO-4-METHOXYTRICYCLO[7.3.1.0<sup>2,7</sup>]TRIDECA-2(7)3,5-TRIENE  
 $C_{16}H_{21}NO_2$

P. MURRAY-RUST, J. MURRAY-RUST and D. MIDDLEMISS, 1982. Acta Cryst., B38, 311-312.

Orthorhombic, Pbca,  $a = 10.43$ ,  $b = 31.40$ ,  $c = 8.67$  Å,  $Z = 8$ . Mo radiation,  $R = 0.044$  for 1070 reflexions.

The molecules (Fig. 1) are held together in the crystal by hydrogen bonds with  $O(2) \dots N(1')$  2.98 Å. The methoxy group is coplanar with the benzene ring and otherwise the geometry is much as expected, with the central bicyclo[3.3.1]nonane system having a sofa [C(7)]-chair [C(3)] conformation.

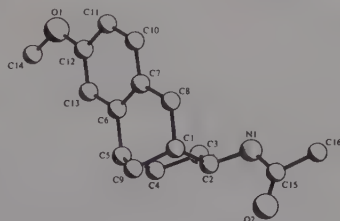


Fig. 1. A perspective view of the  $C_{16}H_{21}NO_2$  molecule.

2-PIPERIDINOTRICYCLO[5.2.2.0<sup>2,6</sup>]UNDECAN-9-ONE  
 $C_{16}H_{25}NO$

K.A. NIRMALA and D.S.S. GOWDA, 1982. Acta Cryst., B38, 839-842.

Monoclinic, Cc,  $a = 24.28$ ,  $b = 8.76$ ,  $c = 14.03$  Å,  $\beta = 108.00^\circ$ ,  $D_m = 1.18$ ,  $Z = 8$ . Cu radiation,  $R = 0.063$  for 1165 reflexions (photographic data).

Bond distances and angles in the two independent molecules (Fig. 1) in the asymmetric unit are similar and are in good agreement with expected values. There are no short intermolecular contacts.

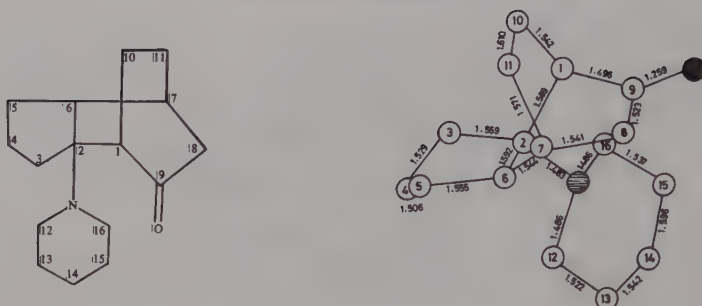


Fig. 1.  $C_{16}H_{25}NO$ : bond lengths (averaged for molecules I and II).

6,6'-DIMETHYL-7,7'-BIS(BICYCLO[3.1.1]HEPTYL)

$C_{16}H_{26}$

P.G. GASSMAN and K.D. OLSON, 1982. *J. Am. Chem. Soc.*, **104**, 3740-3742.

Monoclinic,  $P2_1/c$ ,  $a = 11.045$ ,  $b = 10.593$ ,  $c = 12.741$  Å,  $\beta = 115.21^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.068$  for 1202 reflexions.

The molecule was found to possess the symmetrical structure shown in Fig. 1 (approximate  $C_{2h}$  symmetry). No unusual structural features were noted.

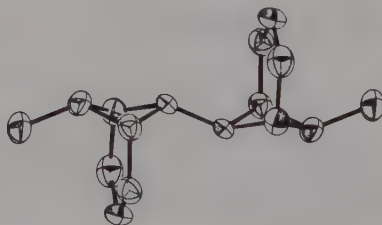


Fig. 1. The  $C_{16}H_{26}$  molecule.

(-)-(Z)-2-(p-CHLOROBENZYLIDENE)BORNAN-3-ONE

$C_{17}H_{19}ClO$

R. ROQUES, C. BERTRAND, F. LABRUYÈRE, J.P. DECLERCQ and G. GERMAIN, 1982. *Acta Cryst.*, **B38**, 646-648.

Orthorhombic,  $P2_12_12_1$ ,  $a = 11.587$ ,  $b = 11.234$ ,  $c = 11.021$  Å,  $Z = 4$ . Cu radiation,  $R = 0.071$  for 1126 reflexions.

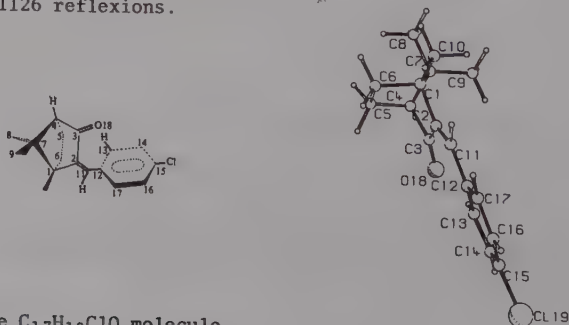


Fig. 1. The  $C_{17}H_{19}ClO$  molecule.

The conjugated system  $O=C-C=C$  is slightly skewed in the sense of a left-handed helix with the  $C(11)-C(2)-C(3)-C(18)$  torsion angle  $-3^\circ$ . The  $C(2)-C(11)-C(12)-C(13)$  angle is  $+7^\circ$ , and the distance between  $O(18)$  and  $H(C13)$  is  $2.03 \text{ \AA}$ . The molecule adopts the *Z* configuration (Fig. 1) as shown by other studies.

exo-10-CHLORO-exo-8-(TOSYLOXY)TRICYCLO[4.3.1.0<sup>1,6</sup>]DECANE

$C_{17}H_{21}ClO_3S$

L.A.M. TURKENBURG, W.H. de WOLF, F. BICKELHAUPT, C.H. STAM and M. KONIJN, 1982. *J. Am. Chem. Soc.*, **104**, 3471-3476.

Monoclinic,  $P2_1/c$ ,  $a = 6.459$ ,  $b = 22.497$ ,  $c = 11.568 \text{ \AA}$ ,  $\beta = 94.92^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.057$  for 2183 reflexions.

The structure is shown in Fig. 1. Bond lengths and angles ( $\sigma = 0.003-0.012 \text{ \AA}$ ,  $0.2-0.8^\circ$ ) are generally as expected. There is possible conformational disordering of the  $C1-C5$  ring involving atoms  $C3$  and  $C4$ .

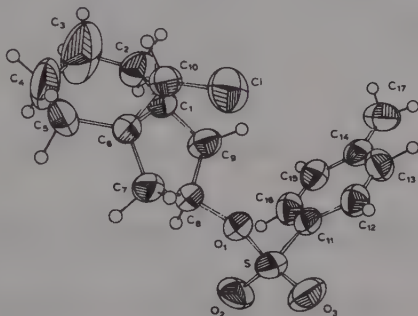


Fig. 1. The  $C_{17}H_{21}ClO_3S$  molecule.

2-(2,6-DIMETHOXYPHENYL)-2,5-DIMETHYLBICYCLO[3.2.1]OCTANE-6,8-DIONE

$C_{18}H_{22}O_4$

P.S. MURTHY, K. VENKATESAN, S.M. REDDY and T.R. KASTURI, 1982. *Acta Cryst.*, **B38**, 2055-2057.

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.343$ ,  $b = 11.251$ ,  $c = 19.357$ ,  $D_m = 1.20$ ,  $Z = 4$ . Mo radiation,  $R = 0.052$  for 1053 reflexions.

The configuration at  $C(2)$  is 2-exo-methyl-2-endo-(2,6-dimethoxyphenyl) i.e. the  $C(2)$  methyl and bridge-keto group are syn. The five-, six-, and seven-membered rings of the bicyclooctane moiety are respectively in the distorted envelope, chair and boat conformations (Fig. 1).

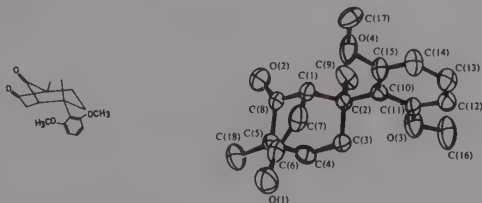


Fig. 1. The  $C_{18}H_{22}O_4$  molecule.



## 9,9'-BIBICYCLO[3.3.1]NONYLIDENE

 $C_{18}H_{28}$ W.H. WATSON, D.A. GROSSIE and I.F. TAYLOR, 1982. *Acta Cryst.*, **B38**, 3159-3161.

Monoclinic,  $C2/m$ ,  $a = 14.257$ ,  $b = 15.015$ ,  $c = 6.895 \text{ \AA}$ ,  $\beta = 93.21^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.045$  for 790 reflexions.

Two independent molecules sit on sites of  $2/m$  symmetry. In one molecule the twofold axis is along the  $C=C$  bond and in the other it is perpendicular to this bond. Both molecules have all-chair conformation with transannular  $H\dots H$  contacts of  $1.99(3)$  and  $1.81(8) \text{ \AA}$ .

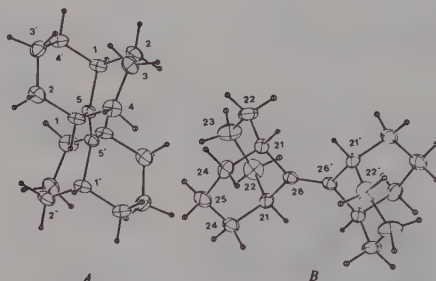


Fig. 1. Drawings of the two  $C_{18}H_{28}$  molecules.

exo-9-p-NITROBENZOYLPENTACYCLO[6.2.1.1<sup>3,6</sup>.0<sup>2,7</sup>.0<sup>4,10</sup>]DODECAN-5-ONE  
 $C_{19}H_{17}NO_5$

R. CERNIK, G.-A. CRAZE, O.S. MILLS and I. WATT, 1982. *J. Chem. Soc. Perkin II*, 361-367.

Monoclinic,  $P2_1/c$ ,  $a = 7.321$ ,  $b = 17.942$ ,  $c = 11.866 \text{ \AA}$ ,  $\beta = 96.07^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.048$  for 1303 reflexions.

The endocyclic bond angle at the carbonyl function,  $C(13)-C(18)-C(17)$  (Fig. 1), is  $104.0$ , while the exocyclic angles are  $C(17)-C(18)-O(5)$   $127.9$  and  $C(13)-C(18)-O(5)$   $127.2^\circ$ . The  $C(18)-O(5)$  bond length is  $1.203 \text{ \AA}$ . There is a significant deviation from planarity in the carbonyl function with  $C(18)$  displaced towards  $C(12)$  by  $0.074 \text{ \AA}$  out of the plane of  $O(5)$ ,  $C(17)$  and  $C(13)$ . The separation of  $C(12)$  and  $C(18)$  is  $2.583 \text{ \AA}$ . The distortion at  $C(18)$  is consistent with NMR evidence of incipient hydride transfer from  $C(12)$  to the carbonyl carbon. There are no short intermolecular contacts.

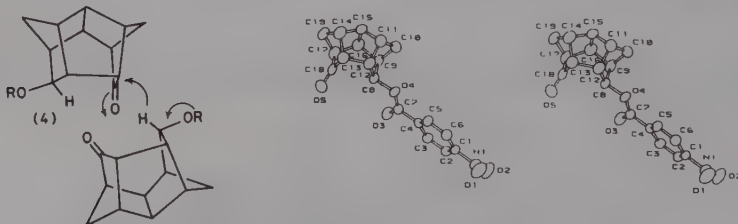


Fig. 1. Molecular skeleton and structure of  $C_{19}H_{17}NO_5$ .

## (E)-2-ETHYNYL-2-METHOXY-5-PHENYLADAMANTANE

 $C_{19}H_{22}O$ 

S.Y. LIN, M. IONOV, Y. OKAYA, D.M. CHIOU and W.J. LE NOBLE, 1982. Acta Cryst., B38, 1666-1669.

Orthorhombic,  $Pc2_1n$ ,  $a = 6.972$ ,  $b = 20.214$ ,  $c = 21.025$  Å,  $D_m = 1.199$ ,  $Z = 8$ . Cu radiation,  $R = 0.057$  for 2065 reflexions.

The two independent molecules in the asymmetric unit (Fig. 1) are arranged around pseudo inversion centres. The  $OCH_3$  group is anti with respect to the phenyl plane, with the methoxy bond almost parallel to the  $O(1)-C(2)-C(1)-C(10)$  plane. The dihedral angles between the approximate mirror planes through  $C(2), C(5), C(6), C(7)$  and the phenyl planes are  $87$  and  $80^\circ$  in the two molecules. The  $C(1e)-C(2e)$  bond lengths are  $1.158(4)$  and  $1.186(4)$  Å.

Fig. 1. The  $C_{19}H_{22}O$  molecule and an ORTEP plot.

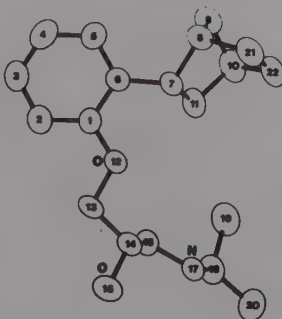
## 1-(2-exo-BICYCLO[2.2.1]HEPT-2-YL-PHENOXY)-3-ISOPROPYLAMINO-2-PROPANOL HYDROCHLORIDE

 $C_{19}H_{30}ClNO_2$ 

J.-M. LEGER, J.-C. COLLETER and A. CARPY, 1982. Cryst. Struct. Comm., 11, 1363-1368.

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.204$ ,  $b = 13.829$ ,  $c = 19.575$  Å,  $Z = 4$ . Cu radiation,  $R = 0.038$  for 1993 reflexions.

A view of this  $\beta$ -blocker is given in Fig. 1. The molecule is the  $\alpha S, 2S$  enantiomer. The centers which have most frequently been implied in the interaction between  $\beta$ -blockers and their adrenoceptors are the quaternary N, the alcoholic OH and the aromatic ring. The following distances are given:  $D_1$ , the distance between  $\pi$  (middle of  $C(1)-C(2)$  and  $O(15)$ ;  $D_2$ , the distance between  $\pi$  and  $N^+(17)$ ;  $D_3$ , the distance between  $O(15)$  and  $N^+(17)$ ;  $D_4$ , the distance between  $O(15)$  and the plane of the aromatic ring;  $D_5$ , the distance between  $N^+(17)$  and the plane of the aromatic ring ( $D_1$  4.88,  $D_2$  5.76,  $D_3$  3.13,  $D_4$  0.30,  $D_5$  1.68 Å).

Fig. 1. A view of the  $C_{19}H_{30}NO_2^+$  ion.

endo,endo-7,8-DIPHENYLBICYCLO[4.1.1]OCTA-2,4-DIENE

$C_{20}H_{18}$

K.L. CHASEY, L.A. PAQUETTE and J.F. BLOUNT, 1982. J. Org. Chem., 47, 5262-5270.

Orthorhombic, Pcab,  $a = 10.489$ ,  $b = 10.677$ ,  $c = 26.524$  Å,  $Z = 8$ . Cu radiation,  $R = 0.040$  for 1594 reflexions.

The conjugated diene moiety (Fig. 1) acts as an almost planar belt about the cyclobutane ring. The dihedral angles between the 3-atom planes of the cyclobutane ring are  $22.3$ - $22.6^\circ$ . Although the opposed C1-C4 ( $1.554$  Å) and C2-C3 ( $1.555$  Å) bonds of the four-membered ring compare favorably in length with the average value observed for cyclobutanes ( $1.550$  Å), the alternate two bonds represented by C1-C2 ( $1.572$  Å) and C3-C4 ( $1.569$  Å) are significantly longer. These unexpected characteristics provide for an alternating short bond-long bond arrangement.

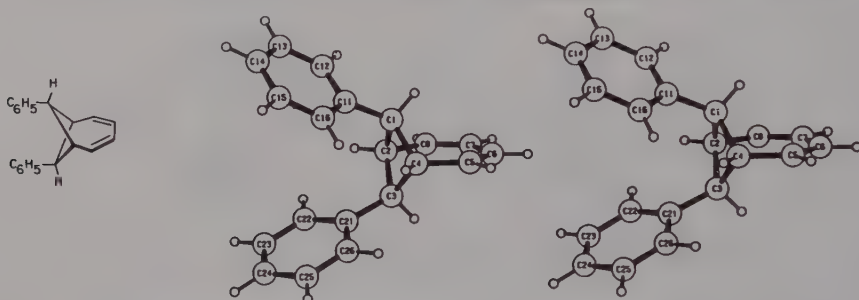


Fig. 1. The  $C_{20}H_{18}$  molecule and stereoview.

4-exo-(2,4-DINITROPHENYLTHIO)-anti-6-PERCHLORYLOXY-9,10-cis-endo-DIMETHOXYCARBONYL-TETRACYCLO[5.3.0.0.2<sup>b</sup>,<sup>b</sup>.0<sup>3,8</sup>]DECANE

$C_{20}H_{19}ClN_2O_{12}S$

N.S. ZEFIROV, A.S. KOZ'MIN, V.V. ZHDANKIN, V.N. KIRIN, K.A. POTEKHIN and Yu.T. STRUCHKOV, 1982. Cryst. Struct. Comm., 11, 1921-1924.

Monoclinic, Cc,  $a = 17.885$ ,  $b = 13.678$ ,  $c = 9.149$  Å,  $\beta = 95.27^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.04$  for 1681 reflexions.

The analysis establishes the tetracyclo-decane structure for this molecule (Fig. 1), and the anti-configuration for the perchloryloxy group.

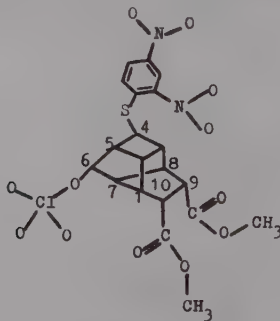


Fig. 1. The  $C_{20}H_{19}ClN_2O_{12}S$  molecule.

1,3-DIMETHYLTETRACYCLO[5.4.0.0<sup>2,4</sup>.0<sup>3,8</sup>]UNDECA-5,10-DIENYL-2-CARBINOL p-BROMOBENZOATE  
 $C_{21}H_{21}BrO_2$

H. KOYAMA and K. OKADA, 1982. Cryst. Struct. Comm., 11, 665-673.

Triclinic,  $P\bar{1}$ ,  $a = 17.010$ ,  $b = 11.004$ ,  $c = 10.466$  Å,  $\alpha = 92.61$ ,  $\beta = 89.61$ ,  $\gamma = 112.24^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.080$  for 3774 reflexions.

The asymmetric unit contains two independent molecules which do not differ significantly (Fig. 1). The compound contains a novel cage system with three six-membered rings and a three-, a five- and a seven-membered ring. Bond distances and angles are as expected.

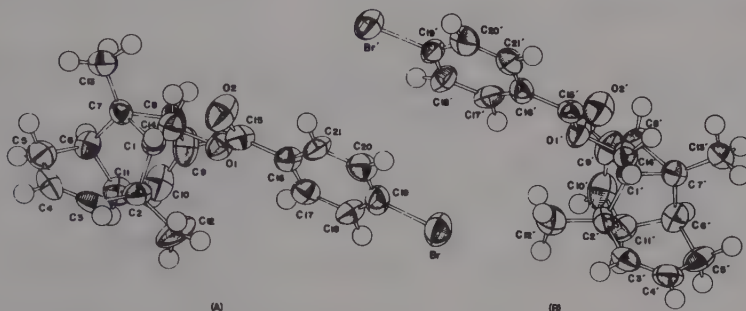


Fig. 1. The two  $C_{21}H_{21}BrO_2$  molecules.

4-CHLORO-2,2-DIMETHYL-7-PHENYLPENTACYCLO[6.3.1<sup>1,8</sup>.1<sup>3,6</sup>.0<sup>1,7</sup>.0<sup>5,12</sup>]TRIDEC-10-ENE  
 $C_{21}H_{23}Cl$

H. MAYR and I.K. HALBERSTADT-KAUSCH, 1982. Chem. Ber., 115, 3479-3515.

Monoclinic,  $P2_1/n$ ,  $a = 8.187$ ,  $b = 15.405$ ,  $c = 25.826$  Å,  $\beta = 91.38^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.08$  for 2253 reflexions.

The structure was confirmed as that shown in Fig. 1 with expected values for the bond lengths and angles.

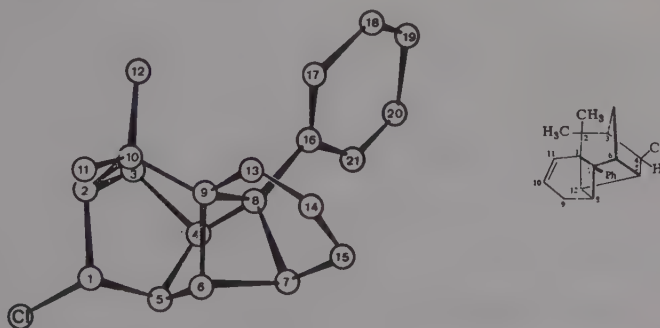


Fig. 1. Perspective view of one of the two independent molecules of  $C_{21}H_{23}Cl$ .

[2](2,5)(7,7,8,8-TETRACYANOQUINODIMETHANO)[2]PARACYCLOPHANE BENZENE SOLVATE  
 $C_{22}H_{14}N_4C_6H_6$  (I)

14,17-DIMETHOXY[2](2,5)(7,7,8,8-TETRACYANOQUINODIMETHANO)[2]PARACYCLOPHANE  
 $C_{24}H_{18}N_4O_2$  (II)

T. MIZUMA, H. MATSUURA, Y. KAI, N. YASUOKA and N. KASAI, 1982. Bull. Chem. Soc. Jpn., 55, 979-984.

I. Triclinic,  $P\bar{1}$ ,  $a = 9.772$ ,  $b = 18.477$ ,  $c = 6.569$  Å,  $\alpha = 90.38^\circ$ ,  $\beta = 107.92^\circ$ ,  $\gamma = 100.32^\circ$ ,  $D_m = 1.240$ ,  $Z = 2$ . Mo radiation,  $R = 0.088$  for 3141 reflexions.

II. Orthorhombic,  $Fdd2$ ,  $a = 25.618$ ,  $b = 31.895$ ,  $c = 9.808$  Å,  $D_m = 1.30$ ,  $Z = 16$ . Mo radiation,  $R = 0.109$  for 1695 reflexions.

The paracyclophane skeletons had the anticipated conformations, with the benzene ring and the six-membered ring of the TCNQ moiety in boat form and the two  $C(CN)_2$  portions of TCNQ bent away from the molecular centre.

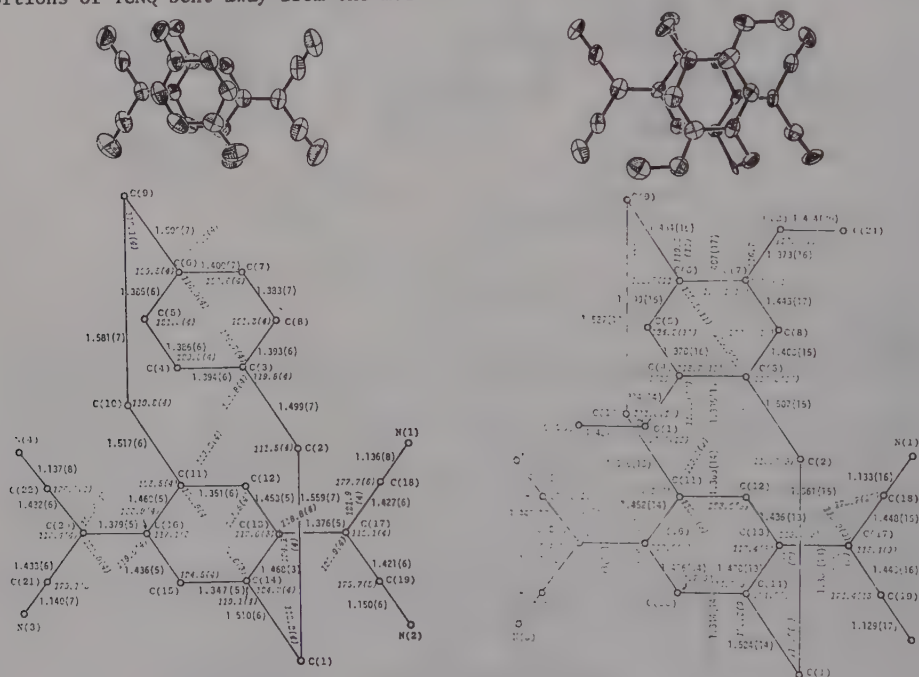


Fig. 1. Views of the paracyclophanes and bond lengths.

[2<sub>4</sub>](1',2',5',6')CYCLOOCTATETRAENYL(1,2,4,5)CYCLOPHANE  
 $C_{22}H_{22}$

A.W. HANSON, 1982. Cryst. Struct. Comm., 11, 1401-1404.

Monoclinic,  $C2/c$ ,  $a = 13.943$ ,  $b = 11.967$ ,  $c = 9.405$  Å,  $\beta = 107.31^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.045$  for 1110 reflexions.

The molecule (Fig. 1) lies about a crystallographic twofold axis and has approximate  $mm2$  symmetry. The cyclooctatetraene molecule has a regular tub conformation with torsion angles  $<0.5^\circ$  for double bonds and  $57.0$  to  $57.1^\circ$  for single bonds. The benzene ring is boat-shaped, with C4 and C7 lying  $0.077$  Å from the plane of the remaining four atoms.

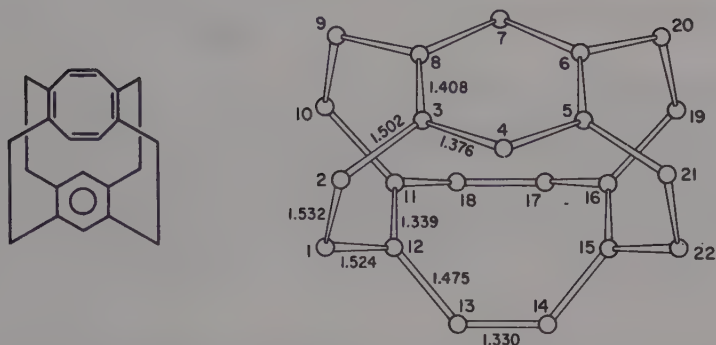


Fig. 1. A view of  $C_{22}H_{22}$  with mean bond lengths (e.s.d.'s are 0.002 to 0.003 Å).

3-BENZYLIDENPENTACYCLO[9.2.1<sup>5,8</sup>.11,11.0<sup>2,10</sup>.0<sup>4,9</sup>]PENTADECANE  
 $C_{22}H_{26}$

$C_6H_5C(H)C_{15}H_{20}$

M. CATELLANI, G.P. CHIUSOLI and P. SGARABOTTO, 1982. *J. Organometal. Chem.*, **240**, 311-319.

Monoclinic,  $P2_1/c$ ,  $a = 13.539$ ,  $b = 6.007$ ,  $c = 20.038$  Å,  $\beta = 94.6^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.042$  for 1526 reflexions.

The molecule (Fig. 1) may be described in terms of three fused fragments: a cyclopentylidene ring and two norbornanes related by a pseudo  $C_2$  axis in the direction of the double bond of the central ring. The bridging C atoms of the two norbornanes are trans with respect to the mean plane of the cyclopentylidene ring. The two norbornane fragments have similar molecular geometry, bond lengths and angles differing by no more than 0.01 Å and  $0.9^\circ$  respectively. The bridgehead angles are  $C(1)-C(14)-C(11)$   $94.7^\circ$  and  $C(5)-C(15)-C(8)$   $94.2^\circ$ . The bond distances and angles in the cyclopentylidene fragment are as expected with the double bond (1.334 Å) localized in the  $C(3)-C(16)$  position. The torsion angle  $C(3)-C(16)-C(17)-C(18)$   $21.7^\circ$ , and the angle  $C(3)-C(16)-C(17)$   $131.1^\circ$  are in agreement with the presence of strain induced by steric hindrance.

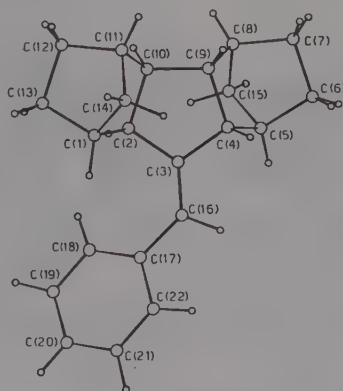


Fig. 1. Projection of  $C_{22}H_{26}$  on the mean plane of the cyclopentylidene ring.



## DIMETHYLMONOSECODODECAHEDRANE

 $C_{22}H_{26}$  (I)

## 1,16-DIMETHYLDODECAHEDRANE

 $C_{22}H_{24}$  (II)

G.G. CHRISTOPH, P. ENGEL, R. USHA, D.W. BALOGH and L.A. PAQUETTE, 1982. J. Am. Chem. Soc., 104, 784-791.

I. Monoclinic,  $P2_1/m$ ,  $a = 7.858$ ,  $b = 10.618$ ,  $c = 8.898$  Å,  $\beta = 91.5^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.102$  for 1532 reflexions.

II. Orthorhombic,  $Pbca$ ,  $a = 7.361$ ,  $b = 13.792$ ,  $c = 13.367$  Å,  $Z = 4$ . Mo radiation,  $R = 0.114$  for 1918 reflexions.

Both molecules are shown in Fig. 1. I possesses approximate  $C_{2v}$  (and exact  $C_s$ ) symmetry with the twofold axis bisecting the missing framework bond. This latter nonbonded C...C distance is 3.030(8) Å, resulting in substantial bond angle distortions of the framework in the vicinity of the missing bond and the flexure of the cyclopentane ring at the gap. (For I: C-C 1.527(6)-1.567(5), mean C-H 1.025(12) Å). The stresses are almost completely relieved in II which possesses approximate  $D_{3d}$  (exact  $C_i$ ) symmetry. The deviations from pure dodecahedral ( $I_h$ ) symmetry induced by the axial substituents are very slight, amounting to only a 0.062(4) Å elongation in the threefold direction. (For II: C-C 1.541(3)-1.551(2) Å for framework, C-Me 1.533(3), mean C-H 0.98 Å).

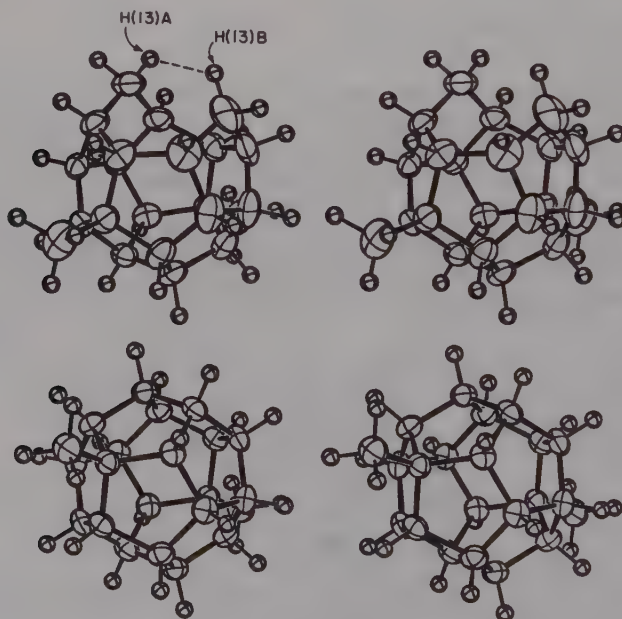


Fig. 1. Stereoviews of  $C_{22}H_{26}$  (top) and  $C_{22}H_{24}$  (bottom).

## 1,2,3,4-TETRACHLORO-9-(2-OXOPROPYL)TRIPTYCENE

 $C_{23}H_{14}Cl_4O$ 

N. NOGAMI, M. OKI, S. SATO and Y. SAITO, 1982. Bull. Chem. Soc. Jpn., 55, 3580-3585.

Monoclinic,  $P2_1/a$ ,  $a = 30.812$ ,  $b = 8.304$ ,  $c = 15.103$  Å,  $\beta = 97.19^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.048$  for 6067 reflexions.

The two independent molecules in the asymmetric unit are the *ap* and  $\pm sc$  rotational isomers about the C9-C<sub>pr</sub> bond (Fig. 1). The acetyl group in the 9-substituent takes an O-inside, relative to the triptycene skeleton, conformation in both forms. The benzene rings are deformed by the steric effect to form boats. The 2-oxopropyl groups in both forms were planar within the experimental error but the C2-C1 bond in the 2-oxopropyl group was appreciably shorter in the  $\pm sc$  conformation than that in the *ap*. The results are discussed on the ground of steric repulsion and the existence of the interaction between the C=O and the Cl groups which are located only 3.035 Å apart in the  $\pm sc$  form.

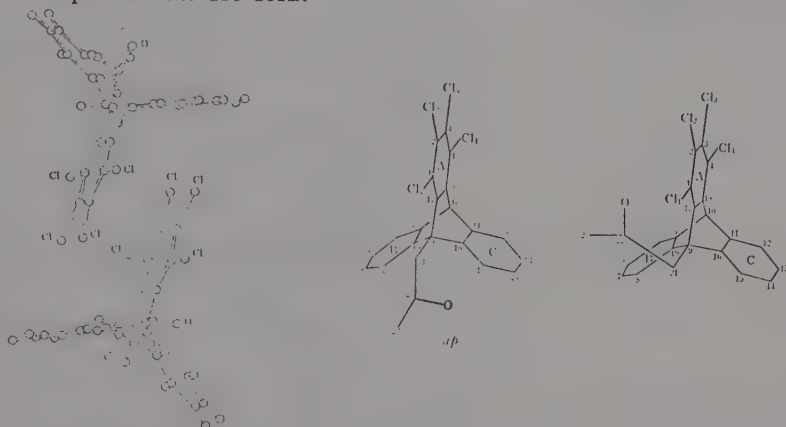


Fig. 1. The  $C_{23}H_{14}Cl_4O$  molecules and schematic drawings.

2,2-DIPHENYLTRICYCLO[4.4.1.0<sup>1,4</sup>]UNDECAN-3-ONE  
 $C_{23}H_{24}O$

K.B. BECKER, M.K. HOHERMUTH and G. RIHS, 1982. *Helv. Chim. Acta*, **65**, 235-242.

Monoclinic,  $P2_1$ ,  $a = 8.628$ ,  $b = 15.293$ ,  $c = 6.808$  Å,  $\beta = 97.39^\circ$ ,  $Z = 2$ . [?] radiation,  $R = 0.077$  for 1514 reflexions.

The cyclobutane ring (Fig. 1) is puckered. The dihedral angle between the plane containing the carbonyl group (C(2), C(3), C(4)) and the plane containing the fully substituted bridgehead (C(1), C(2), C(4)) is  $32^\circ$ . The carbonyl group is not exactly planar. The displacement of the O-atom from the (C(2), C(3), C(4))-plane is 0.132 Å and the O-atom lies on the same side of the plane as C(1). Bond lengths and angles have normal values.

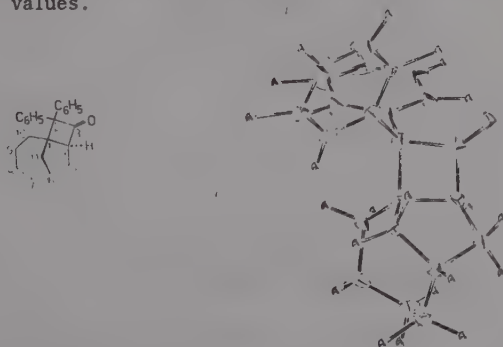


Fig. 1. The  $C_{23}H_{24}O$  structure.

## 9-(FORMYLMETHYL)-1,4-DIMETHYLTRIPTYCENE

 $C_{24}H_{20}O$ 

M. OKI, G. IZUMI, G. YAMAMOTO and N. NAKAMURA, 1982. Bull. Chem. Soc. Jpn., 55, 159-166.

Orthorhombic,  $Pbca$ ,  $a = 15.374$ ,  $b = 9.801$ ,  $c = 23.112$  Å,  $Z = 8$ . Cu radiation,  $R = 0.070$  for 2575 reflexions.

The formyl group adopts an O-outside conformation (Fig. 1) and steric stress caused by substituents at the C(1) and C(9) positions is released by deformation throughout the molecule.

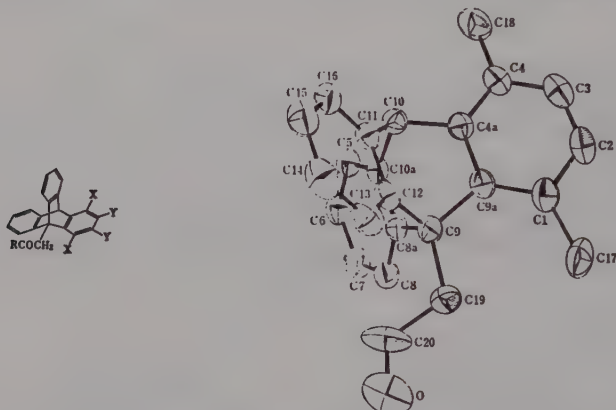


Fig. 1. The  $C_{24}H_{20}O$  molecule ( $X = Me$ ,  $Y = R = H$ ).

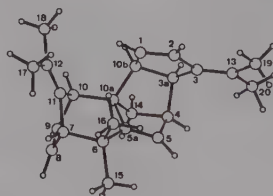
## 6,6-DIMETHYLFULVENE TRIMER

 $C_{24}H_{30}$ 

B. UEBERSAX, M. NEUENSCHWANDER and P. ENGEL, 1982. Helv. Chim. Acta, 65, 89-104.

Triclinic,  $P\bar{1}$ ,  $a = 8.004$ ,  $b = 9.443$ ,  $c = 13.06$  Å,  $\alpha = 94.67^\circ$ ,  $\beta = 96.28^\circ$ ,  $\gamma = 100.92^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.03$  for 2593 reflexions.

The analysis established the structure of this trimer of 6,6-dimethylfulvene to be as shown in Fig. 1. The molecule is quite strained and the angle C(4)-C(14)-C(10a) is only  $94.1^\circ$ .



The structure consists of two independent centrosymmetric molecules with nearly 2/m symmetry. The two six-membered rings are shifted by 0.62 Å with respect to one another (Fig. 1); this produces a torsion angle of 15° for the dimethylene bridges (C-C 1.563(8) Å). The tetramethylbenzene rings are folded along the C(5)...C(11) vector to a V-shape (Fig. 1) with a dihedral angle of 17.5°. The mean distance between the rings is 3.16 Å.

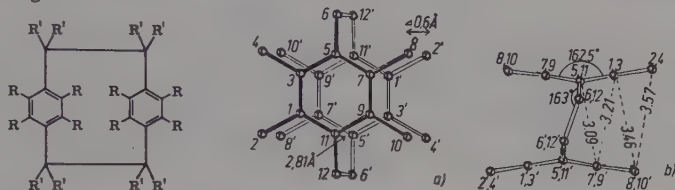


Fig. 1. Views of  $C_{24}H_{32}$  ( $R = CH_3$ ,  $R' = H$ ) perpendicular and parallel to the benzene rings.

[2] (2,5) (7,7,8,8-TETRACYANOQUINODIMETHANO) [2] (1,4)NAPHTHALENOPHANE  
C<sub>26</sub>H<sub>16</sub>N<sub>4</sub>

T. MIZUMA, K. MIKI, Y. KAI, N. TANAKA and N. KASAI, 1982. *Bull. Chem. Soc. Jpn.*, **55**, 2026-2028.

Monoclinic, Cc,  $a = 9.770$ ,  $b = 15.176$ ,  $c = 13.299$  Å,  $\beta = 98.43^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.035$  for 1217 reflexions.

In this molecule (Fig. 1), the [2,2]paracyclophane skeleton has the expected structure; a half of the naphthalene moiety has a boat-form, the six-membered ring of the TCNQ moiety also has a boat-form, and two C(CN)<sub>2</sub> portions in the TCNQ moiety are bent out and slightly twisted from the six-membered ring. The other half of the naphthalene ring free from the bridging is planar and is slightly bent out from the [2,2]paracyclophane skeleton.

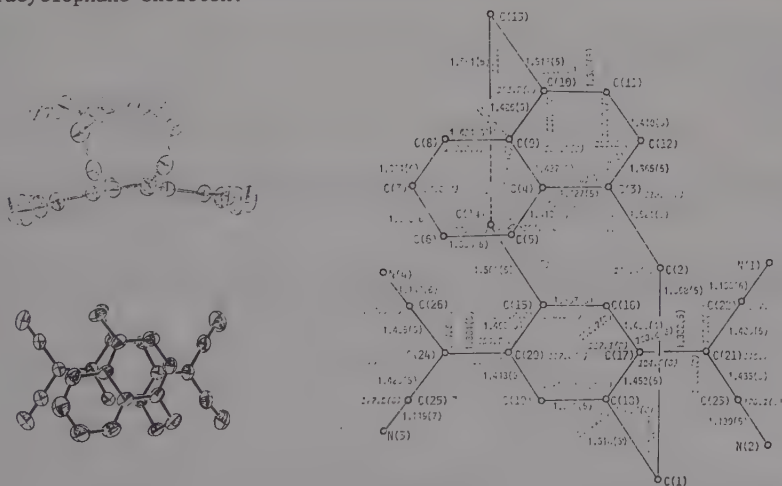


Fig. 1. Views of the  $C_{26}H_{16}N_4$  molecule and dimensions.

9,13-ETHENO-6,8-DIETHYL-2,3,4-NAPHTHO TRICYCLO[6.5.0.0<sup>1,5</sup>]TRIDECA-5,11-DIEN-7-ONE  
C<sub>26</sub>H<sub>24</sub>O

K. HARANO, M. YASUDA and K. KANEMATSU, 1982. J. Org. Chem., 47, 3736-3743.  
Cryst. Struct. Comm., 11, 337-341.

Monoclinic, P<sub>2</sub><sub>1</sub>/a, a = 15.345, b = 11.646, c = 10.626 Å, β = 92.979°, D<sub>m</sub> = 1.532, Z = 4. Mo radiation, R = 0.051 for 1497 reflexions.

The analysis confirms (Fig. 1) the syn-endo [2 + 4] π structure with a syn orientation of the carbonyl group to the methylene group. The ring system is moderately strained: interbond angles at the sp<sup>2</sup> carbons (C1-C2-C3 117.7°; C4-C5-C6 124.0; C5 C6 C7 127.2°) deviate by 2.3, 4.0 and 7.2°, respectively, from the unstrained angles of 120°. The sp<sup>3</sup> carbons show similar deviations from the tetrahedral angle of 109.5°. The C4-C12, C1-C13, and C12-C13 bond lengths of 1.59, 1.57, and 1.57 Å are longer than the usual value of 1.54 Å.

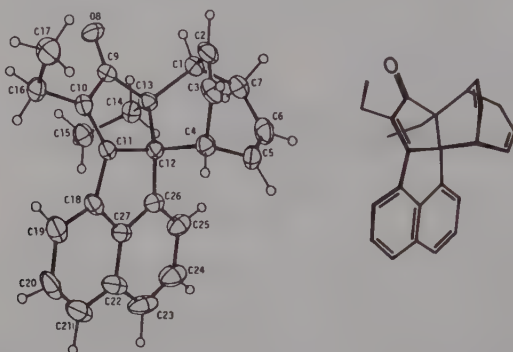


Fig. 1. The C<sub>26</sub>H<sub>24</sub>O molecule.

(DL)-(1R,6S)-HEXACYCLO[10.5.1.0<sup>1,14</sup>.0<sup>6,17</sup>.0<sup>9,17</sup>.0<sup>11,13</sup>]OCTADECA-2,4,7,9,15-PENTAENE-2,3,10,16-TETRARCOXYLIC ACID TETRAMETHYL ESTER  
C<sub>26</sub>H<sub>24</sub>O<sub>8</sub>

K.-H. LEHR, D. HUNKLER, E. HADICKE and H. PRINZBACH, 1982. Chem. Ber., 115, 1857-1874.

Monoclinic, P<sub>2</sub><sub>1</sub>/c, a = 10.640, b = 8.471, c = 25.670 Å, β = 103.55°, Z = 4. Cu radiation, R = 0.040 for 2596 reflexions.

The structure was established as that shown in Fig. 1 with normal values for bond lengths and angles. The conformations of the six rings is also shown in Fig. 1.

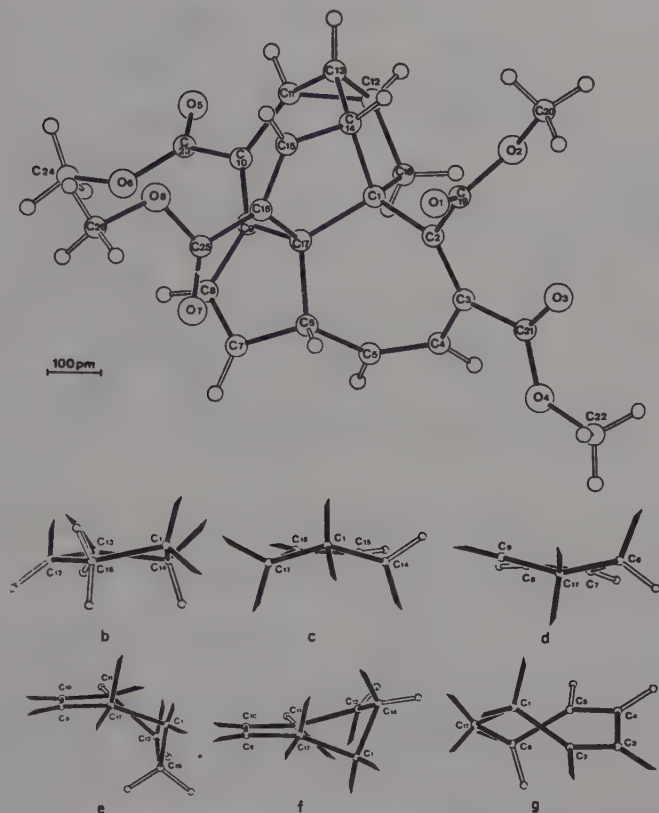


Fig. 1. The molecular structure of  $C_{26}H_{24}O_8$  and the ring conformations.

TETRAMETHYL anti-9,9'-BI((1 $\alpha$ ,2 $\beta$ ,5 $\beta$ ,6 $\alpha$ )TRICYCLO[4.2.1.0<sup>2,5</sup>]NONA-3,7-DIENYLIDENE)-3,3',4,4'-TETRACARBOXYLATE

$C_{26}H_{24}O_8$  (I)

DIMETHYL anti-10-(3,4-BIS(METHOXYCARBONYL)(1 $\alpha$ ,2 $\beta$ ,5 $\beta$ ,6 $\alpha$ )TRICYCLO[4.2.1.0<sup>2,5</sup>]NONA-3,7-DIEN-9-YLIDENE)-8-OXA-(1 $\alpha$ ,2 $\beta$ ,5 $\beta$ ,6 $\beta$ ,9 $\beta$ )-TETRACYCLO[4.3.1.0<sup>2,5</sup>.0<sup>7,9</sup>]DEC-3-EN-3,4-DICARBOXYLATE

$C_{26}H_{24}O_9$  (II)

K.-H. LEHR, J. WERP, H. BINGMANN, C. KRUGER and H. PRINZBACH, 1982. Chem. Ber., **115**, 1835-1856.

I. Monoclinic,  $P2_1/c$ ,  $a = 9.766$ ,  $b = 9.021$ ,  $c = 13.186$  Å,  $\beta = 74.38^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.045$  for 1482 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 9.833$ ,  $b = 8.980$ ,  $c = 13.160$  Å,  $\beta = 106.00^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.061$  for 2066 reflexions.

The two molecules were established as having the centrosymmetric structures shown in Fig. 1 with normal values for bond lengths and angles. To preserve the crystallographically imposed overall  $C_i$  symmetry II is disordered at 0(5).



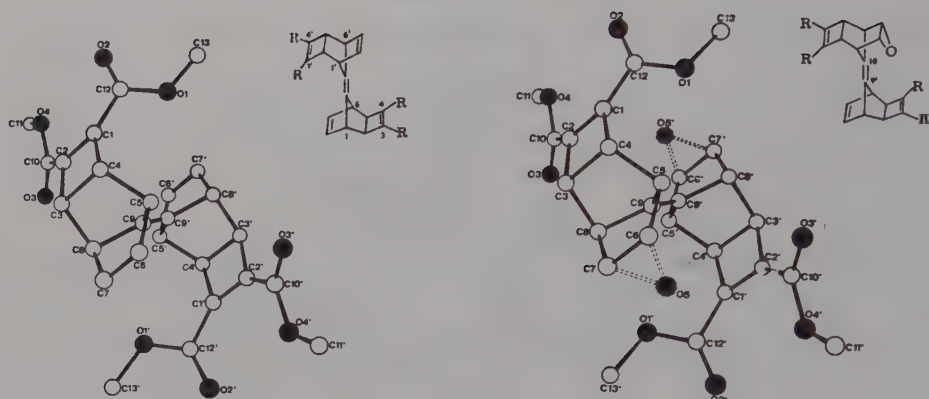


Fig. 1. The structures of  $C_{26}H_{24}O_8$  (left) and  $C_{26}H_{24}O_9$  (right) ( $R = CO_2CH_3$ ).

BIS(9-TRYPTYCYL) KETONE ACETONE SOLVATE

$C_{41}H_{26}O, 0.33(C_3H_6O)$  (I)

BIS(9-TRYPTYCYL)METHANE DICHLOROMETHANE SOLVATE HYDRATE

$C_{41}H_{28}, 0.5(CH_2Cl_2), 0.5(H_2O)$

C.A. JOHNSON, A. GUENZI, R.B. NACHBAR, Jr., J.F. BLOUNT, O. WENNERSTROM and K. MISLOW, 1982. J. Am. Chem. Soc., 104, 5163-5168.

I. Monoclinic,  $A2/a$ ,  $a = 16.760$ ,  $b = 19.885$ ,  $c = 28.738 \text{ \AA}$ ,  $\beta = 111.56^\circ$ ,  $Z = 12$ .  
Cu radiation,  $R = 0.056$  for 4780 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 19.717$ ,  $b = 20.001$ ,  $c = 16.590 \text{ \AA}$ ,  $\beta = 112.23^\circ$ ,  $Z = 8$ .  
Cu radiation,  $R = 0.093$  for 3217 reflexions.

There are two independent molecules in the asymmetric unit of each structure. In I, one set of molecules is in a general position and the other set is located on a twofold axis while both independent molecules in II occupy general positions (see Fig. 1). The molecules are tightly meshed static gear systems. The values of the C-CO-C angles ( $128.5$  and  $128.8^\circ$ ) and of the C-CO bond lengths ( $1.531$ - $1.566 \text{ \AA}$ ) in  $C_1$ - and  $C_2$ -I, and of the C-CH<sub>2</sub>-C bond angle ( $129.3^\circ$ ) and C-CH<sub>2</sub> bond lengths ( $1.532$ ,  $1.578 \text{ \AA}$ ) in  $C_1$ -II, indicate that these systems are under considerable internal strain.

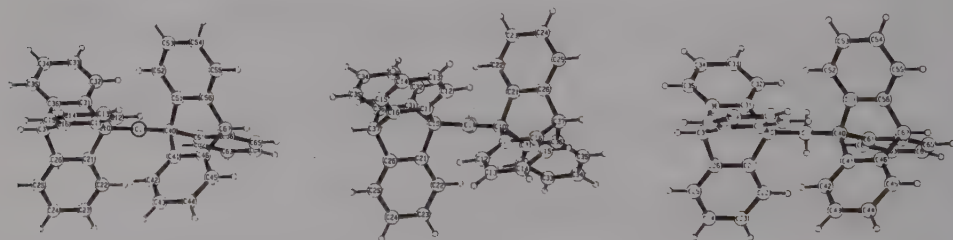
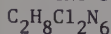


Fig. 1. Views of the two independent molecules of  $C_{41}H_{26}O$  (left and centre) and of one of the  $C_{41}H_{28}$  molecules (right).

## 5-AMINO-3-HYDRAZINO-1,2,4-TRIAZOLE DIHYDROCHLORIDE



G. RECK and M. JUST, 1982. Cryst. Struct. Comm., 11, 1857-1861.

Orthorhombic, Pbcn,  $a = 12.974$ ,  $b = 8.817$ ,  $c = 13.289$  Å,  $Z = 8$ . Mo radiation,  $R = 0.052$  for 971 reflexions.

The dimensions (Fig. 1) are consistent with a delocalization of the  $\pi$ -electrons. There are eight symmetry independent N-H...Cl hydrogen bridges.



Fig. 1. Bond lengths and angles in  $\text{C}_2\text{H}_8\text{Cl}_2\text{N}_6$  ( $\sigma$  0.006 Å and 0.4°).

## IMIDAZOLE



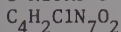
J. EPSTEIN, J.R. RUBLE and B.M. CRAVEN, 1982. Acta Cryst., B38, 140-149.

Monoclinic,  $P2_1/c$ ,  $a = 7.731$  (7.567),  $b = 5.453$  (5.371),  $c = 9.782$  (9.781) Å,  $\beta = 117.28$  (119.05)°,  $Z = 4$ . Mo radiation,  $R = 0.026$  (0.028) for 654 (1892) reflexions (the first set of numbers refers to measurements at 298K, the set in parentheses to 103K).

For previous work see 1. This paper reports on the static charge density of imidazole. The charge density is consistent with a simple electrostatic theory for the strong N-H...N hydrogen bond in the structure.

1. Structure Reports, 43B, 259; 45B, 195.

## 3-NITRO-3'-CHLORO-1H-5,1'-BI(1,2,4-TRIAZOLINE)



G.L. STAROVA, O.V. FRANK-KAMENETSKAYA, O.A. USOV, A.M. KUZ'MIN and M.S. PEVZNER, 1982. Zh. Strukt. Khim., 23-2, 171-173 [J. Struct. Chem., 23, 324-326].

Monoclinic,  $P2_1/n$ ,  $a = 7.411$ ,  $b = 9.090$ ,  $c = 11.914$  Å,  $\gamma = 98.38^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.033$  for 1118 reflexions.

Bond lengths and angles are shown in Fig. 1. The molecule adopts a transoid configuration about the  $\text{N}_1$ -C5 bond. The planar triazole rings are inclined 5° with respect to one another and the  $\text{NO}_2$  is rotated 4° from the plane of the ring to which it is bonded.

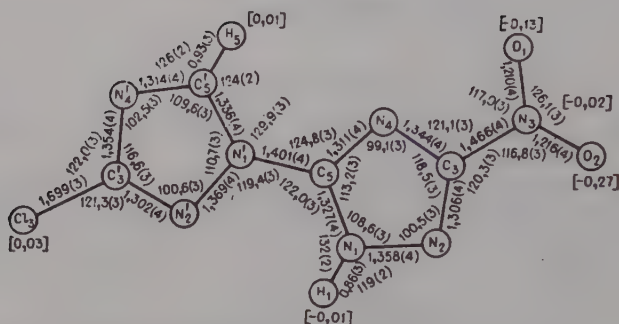
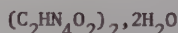
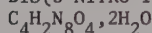


Fig. 1. The  $C_4H_2ClN_7O_2$  molecule.

BIS(3-NITRO-1,2,4-TRIAZOLYL-5) DIHYDRATE



E.V. NIKITINA, G.L. STAROVA, O.V. FRANK-KRAMENETSKAYA and M.S. PEVZNER, 1982. Kristallografiya, 27, 485-488 [Sov. Phys. Crystallogr., 27, 294-296].

Monoclinic,  $P2_1/b$ ,  $a = 3.965$ ,  $b = 14.709$ ,  $c = 8.633$  Å,  $\gamma = 90.26^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.047$  for 629 reflexions.

The triazole rings in the centrosymmetric molecule are planar (Fig. 1), and the terminal H atoms and the N atoms of the nitro groups lie practically in the plane of the rings. The nitro group is tilted by  $4.3^\circ$  from this plane. The water molecule is linked to the triazole ring by a strong hydrogen bond in which the ring hydrogen takes part (N(1)-H(1)...O(3)  $N \dots O$  2.650 Å).

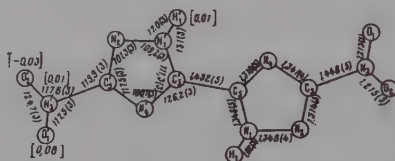
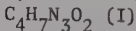
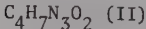


Fig. 1. Bond lengths and angles in  $C_4H_2N_8O_4$ . Figures in square brackets are deviations of atoms from plane of ring.

2,2-AZIRIDINEDICARBOXAMIDE (Form A)



2,2-AZIRIDINEDICARBOXAMIDE (Form B)



S. BRÜCKNER, 1982. Acta Cryst., B38, 2405-2408.

I. Tetragonal,  $P4_12_12$ ,  $a = 12.382$ ,  $c = 30.211$  Å,  $Z = 32$ . Mo radiation,  $R = 0.051$  for 1074 reflexions.

II. Triclinic,  $P1$ ,  $a = 15.829$ ,  $b = 12.381$ ,  $c = 12.391$  Å,  $\alpha = 90.0$ ,  $\beta = 74.3$ ,  $\gamma = 83.1^\circ$ ,  $Z = 16$ . Mo radiation,  $R = 0.070$  for 2534 reflexions.

In the four independent molecules in I, the N atom of the aziridine ring is pyramidal, with mean bond lengths in the ring of 1.463(4) and 1.502(6) Å for N-C and C-C respectively (Fig. 1). No bond lengths and angles are given for II, in which some disorder is present. In both I and II crystal packing may be described in terms of layers, all having the same internal hydrogen-bonded structure, and the two

crystalline forms are characterised by a different stacking of these layers.

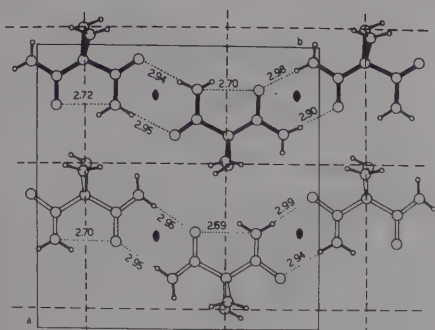
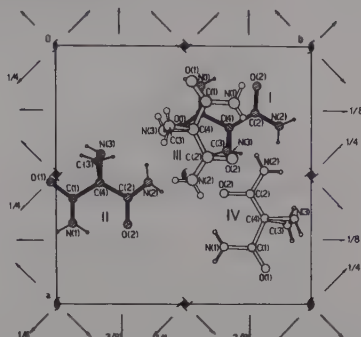
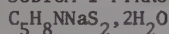


Fig. 1.  $C_4H_7N_3O_2$ : views showing hydrogen bonding and the four independent molecules in form A (I).



SODIUM-1-PYRROLIDINECARBODITHIOATE DIHYDRATE



I. YMÉN, 1982. Acta Cryst., B38, 2671-2674.

Monoclinic,  $P2_1/a$ ,  $a = 12.121$ ,  $b = 5.789$ ,  $c = 14.008$  Å,  $\beta = 98.58^\circ$ ,  $Z = 4$ . Neutron diffraction,  $R = 0.069$  for 1132 reflexions.

In the crystal structure (Fig. 1) the dithiocarbamate ion acts as a monodentate ligand and the  $\text{Na}^+$  ion is surrounded by four water O atoms and two S atoms forming a distorted octahedron. The anion has two short intramolecular H...S contacts, 2.85(1) and 2.84(2) Å, somewhat shorter than the expected van der Waals distance. The S atoms and water molecules form S...H-O hydrogen bonds. The S...H and S...O distances are in the range 2.22(1)-2.36(1) and 3.209(9)-3.308(9) Å respectively. It is concluded that these hydrogen bonds have no detectable effects on the O-H distances.

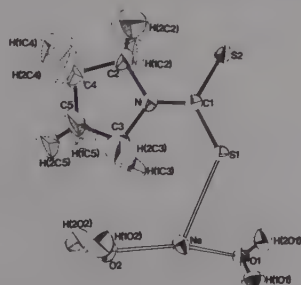
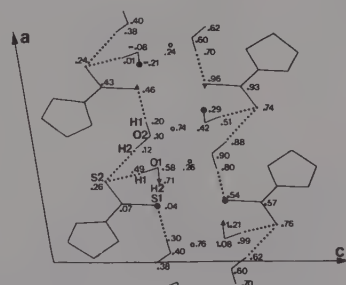
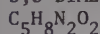


Fig. 1.  $\text{Na}[\text{S}_2\text{CN}(\text{CH}_2)_4] \cdot 2\text{H}_2\text{O}$ : views of the asymmetric unit in the crystal and of the hydrogen-bond system.



## 5,5-DIMETHYLHYDANTOIN



R.E. CASSADY and S.W. HAWKINSON, 1982. *Acta Cryst.*, B38, 1646-1647.

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.2160$ ,  $b = 7.2030$ ,  $c = 13.0050$ ,  $D_m = 1.261$ ,  $Z = 4$ . Mo radiation,  $R = 0.042$  for 777 reflexions.

The almost planar molecules are linked into ribbons by pairs of hydrogen bonds

(N(1)...O(6) 2.870(4) and N(3)...O(6) 2.837(3) Å) between adjacent molecules; O(7) is not hydrogen bonded. Molecular dimensions (Fig. 1) are normal.

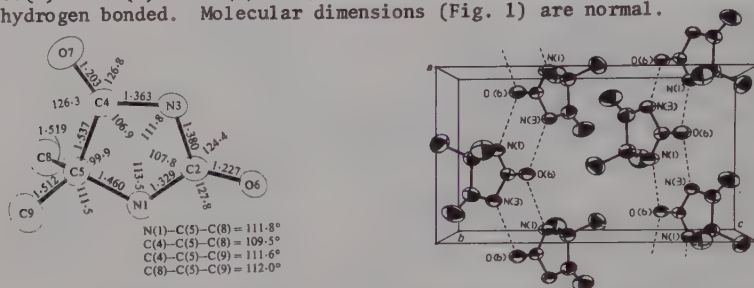


Fig. 1. Bond lengths and angles in 5,5-dimethylhydantoin, and a view of the unit cell.

# (2-OXO-1-PYRROLIDINYL)ACETAMIDE



G. ADMIRAAL, J.C. EIKELENBOOM and A. VOS, 1982. *Acta Cryst.*, **B38**, 2600-2605.

I. Triclinic,  $\bar{P}1$ ,  $a = 6.403$ ,  $b = 6.618$ ,  $c = 8.556$  Å,  $\alpha = 79.85^\circ$ ,  $\beta = 102.39^\circ$ ,  $\gamma = 91.09^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.058$  for 2499 reflexions.

II. Monoclinic,  $P2_1/n$ ,  $a = 6.525$ ,  $b = 6.440$ ,  $c = 16.463$  Å,  $\beta = 92.19^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.064$  for 2435 reflexions.

In both structures (Fig. 1) the planes of the non-H atoms of the pyrrolidone and acetamide groups are almost perpendicular to each other. The molecules form dimers via N-H...O bonds between acetamide groups around an inversion centre and successive dimers are linked together by further N-H...O bonds to form chains. The chains are packed by van der Waals interactions.

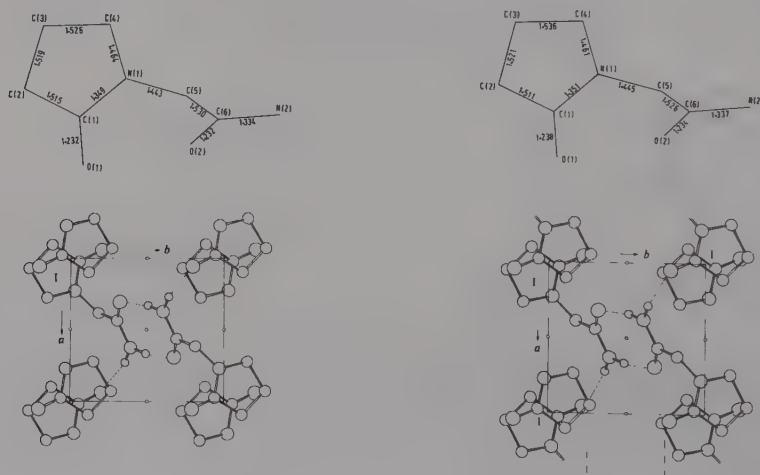


Fig. 1.  $\text{C}_6\text{H}_{10}\text{N}_2\text{O}_2$ : bond lengths and angles and views of the crystal structure (projected on the ab plane) for the triclinic modification (left) and for the monoclinic modification (right).

5,5-DIMETHYL-3-DIMETHYLAMINO-2,4-DIOXO-5-AZONIAIMIDAZOLIDIDE  
 $C_6H_{12}N_4O_2$  (I)

1,3,5-TRIS(DIMETHYLAMINO)-1,3,5-HEXAHYDROTRIAZINE-2,4,6-TRIONE ACETONITRILE SOLVATE  
 $C_{11}H_{21}N_7O_3$  (II)

V. ZURN, W. SCHWARZ, W. ROZDZINSKI and A. SCHMIDT, 1982. *Z. Naturforsch.*, **37b**, 81-90.

I. Orthorhombic,  $Cmc2_1$ ,  $a = 9.262$ ,  $b = 7.191$ ,  $c = 12.755$  Å,  $D_m = 1.33$ ,  $Z = 4$ . Mo radiation,  $R = 0.036$  for 767 reflexions (at 173 K).

II. Rhombohedral,  $R3c$ ,  $a = 9.386$ ,  $c = 29.199$  Å,  $D_m = 1.33$ ,  $Z = 6$ . Mo radiation,  $R = 0.028$  for 805 reflexions (at 173 K).

I (Fig. 1) has  $m$  symmetry and principal bond lengths N(1)-N(2) 1.466(3), N(1)-C(2) 1.534(4), N(1)-C(11) 1.498(2), N(2)-C(1) 1.329(4), C(1)-O(1) 1.237(3), N(3)-C(1) 1.462, N(3)-C(2) 1.331(3), N(3)-N(4) 1.418(3), C(2)-O(2) 1.202(4) Å. II (Fig. 1) has  $C_3$  symmetry (crystallographic) and  $C_{3h}$  within experimental error. Principal bond lengths are N(1)-C(1) 1.400(2), N(1)-N(2) 1.417(1), C(1)-O(1) 1.207(2) Å.

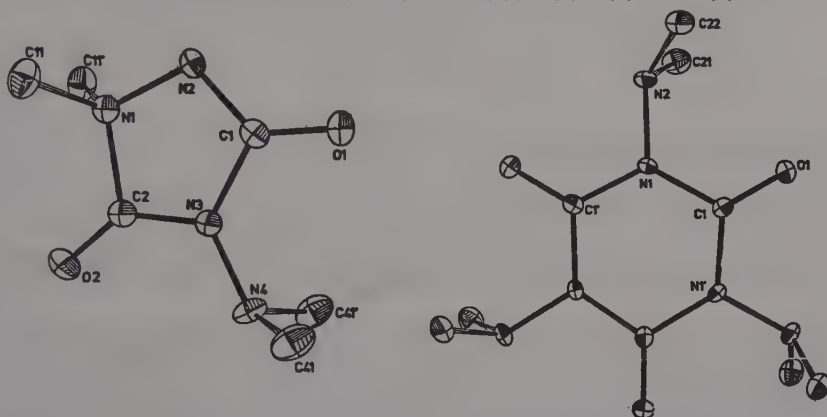


Fig. 1. The structures of  $C_6H_{12}N_4O_2$  (I) (left) and  $C_{11}H_{21}N_7O_3$  (II) (right with the solvate molecule omitted).

3-(1-PYRROLIDINYL)PROPANOIC ACID HEMIHYDRATE  
 $C_7H_{13}NO_2 \cdot 0.5(H_2O)$

M.A. PETERSON, H. HOPE and C.P. NASH, 1982. *Acta Cryst.*, **B38**, 2490-2493.

Monoclinic,  $P2_1/a$ ,  $a = 11.886$ ,  $b = 10.801$ ,  $c = 12.539$  Å,  $\beta = 103.60^\circ$ ,  $D_m = 1.25$  (294 K),  $Z = 8$ . Mo radiation,  $R = 0.0485$  for 2077 reflexions (at 140 K).

The two independent, zwitterionic amino acid molecules form chains, roughly parallel to  $b$ , that are linked by alternating short (N...O 2.649(3) Å) and unevenly bifurcated N-H...O hydrogen bonds (Fig. 1). Adjacent chains are connected in the  $a$  direction by bridging water molecules. One pyrrolidine ring is unusual in that a C atom bonded to the N atom is the most puckered centre.



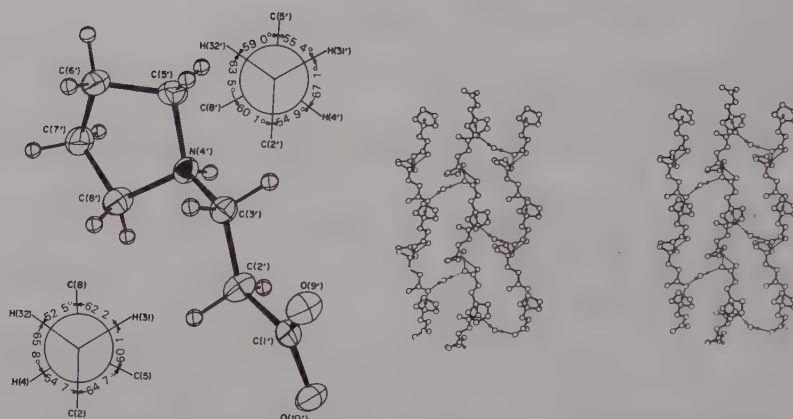


Fig. 1.  $C_7H_{13}NO_2 \cdot 0.5(H_2O)$ : molecular conformation with Newman projections of both molecules down the appropriate  $C(3)-N(4)$  bond (left) and a stereoscopic view of the crystal structure showing the hydrogen bonds (right).

2,4-DINITROBENZALDEHYDE (1H-TETRAZOL-5-YL)HYDRAZONE



D.S.S. GOWDA, R. RUDMAN and K.R. ACHARYA, 1982. Acta Cryst., B38, 2487-2490.

Monoclinic,  $P2_1/n$ ,  $a = 10.991$ ,  $b = 9.056$ ,  $c = 11.706$  Å,  $\beta = 101.50^\circ$ ,  $D_m = 1.60$ ,  $Z = 4$ . Cu radiation,  $R = 0.049$  for 1228 reflexions.

The molecule consists of a 2,4-dinitrobenzaldehyde moiety and a tetrazole moiety bridged by a hydrazone chain (Fig. 1). Each molecule is hydrogen-bonded to three other molecules through four hydrogen-bond linkages (two acceptor and two donor) centred on the tetrazole ring. The entire molecule is approximately planar with the exception of one nitro group which is rotated  $39^\circ$  out of the plane of the benzene ring and has a short C-H...O distance that could indicate a weak intramolecular hydrogen bond.

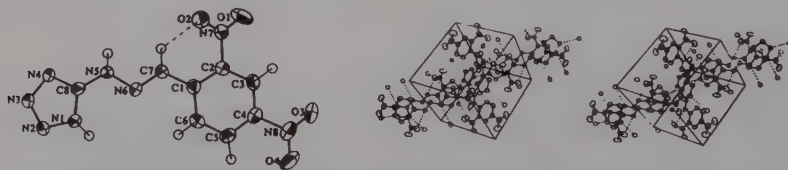


Fig. 1.  $C_8H_6N_8O_4$ : a perspective view of the molecule and a stereoview of the unit cell; hydrogen bonds are shown by broken lines.

(1,3-DIMETHYL-2-IMIDAZOLIDINYLDENE)MALONONITRILE  
 $C_8H_{10}N_4$  (I)

(1,3-DIMETHYL-2-PERHYDOPYRIMIDINYLDENE)MALONONITRILE  
 $C_9H_{12}N_4$  (II)

D. ADHIKESAVALU and K. VENKATESAN, 1982. Acta Cryst., B38, 855-859.

I. Monoclinic,  $P2_1/a$ ,  $a = 7.965$ ,  $b = 16.232$ ,  $c = 7.343$  Å,  $\beta = 113.54^\circ$ ,  $D_m = 1.218$ ,  $Z = 4$ . Cu radiation,  $R = 0.058$  for 1351 reflexions.

II. Orthorhombic,  $P2_1cn$ ,  $a = 7.983$ ,  $b = 8.075$ ,  $c = 14.652$  Å,  $D_m = 1.219$ ,  $Z = 4$ . Mo radiation,  $R = 0.053$  for 538 reflexions.

In both molecules (Fig. 1) the C=C distances are appreciably longer than normal: 1.407(3) Å in I and 1.429(6) Å in II. The steric and push-pull effects result in rotation about the C=C bond, the rotation angles being  $20.2^\circ$  in I and  $31.5^\circ$  in II.

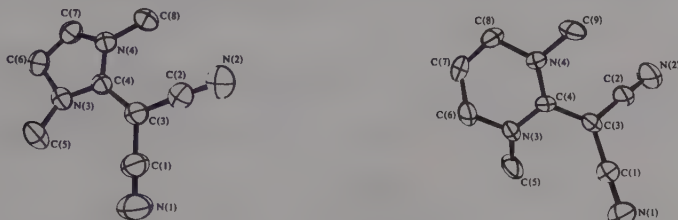


Fig. 1. Perspective views of molecules of  $C_8H_{10}N_4$  (I) (left) and  $C_9H_{12}N_4$  (II) (right).

4-DIBROMOMETHYL-1-HYDROXY-2,2,5,5-TETRAMETHYL-3-IMIDAZOLINE-3-OXIDE  
 $C_8H_{14}Br_2N_2O_2$  (I)

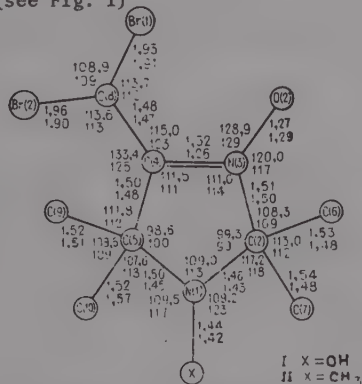
4-DIBROMOMETHYL-1,2,2,5,5-PENTAMETHYL-3-IMIDAZOLINE-3-OXIDE  
 $C_9H_{16}Br_2N_2O$  (II)

Yu.V. GATILOV, M.M. MITASOV, I.A. GRIGOR'EV and L.B. VOLODARSKII, 1982. Zh. Strukt. Khim., 23-6, 91-97 [J. Struct. Chem., 23, 899-904].

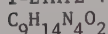
I. Monoclinic,  $P2_1/c$ ,  $a = 15.144$ ,  $b = 7.162$ ,  $c = 11.453$  Å,  $\beta = 107.85^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.056$  for 1431 reflexions.

II. Monoclinic,  $P2_1/n$ ,  $a = 9.616$ ,  $b = 11.741$ ,  $c = 12.083$  Å,  $\beta = 115.62^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.077$  for 1505 reflexions.

Both structures show N(1)-envelope conformations for the heterocyclic ring, N(1) deviating by 0.48 and 0.20 Å from the plane of the other four atoms for I and II respectively. The major difference lies in the orientation of the -CHBr<sub>2</sub> group, the H-C-C-N torsion angles being  $29.5^\circ$  in I and  $177^\circ$  in II. The C(4)=N(3) bond is shorter in II than in I (see Fig. 1)



## 1-ETHYL-4,5-BIS(N-METHYLCARBOYL)IMIDAZOLE [ETHIMIZOLE]



N.B. BROVTSYNA, V.S. FUNDAMENSKII, V.L. GOL'DFARB, K.A. MOSHKOV and Yu.S. BORODKIN, 1982. Zh. Strukt. Khim., 23-2, 92-100 [J. Struct. Chem., 23, 244-252].

Orthorhombic, Fdd2,  $a = 18.496$ ,  $b = 28.666$ ,  $c = 8.301$  Å,  $D_m = 1.25$ ,  $Z = 16$ . Mo radiation,  $R = 0.039$  for 1132 reflexions.

The molecule (Fig. 1) exists in a strained conformation with a strong intramolecular N-H...O hydrogen bond ( $N...O$  2.696(5) Å). The five-membered ring is planar within experimental error and bond distances ( $\sigma$  0.003-0.006 Å) are normal. Molecules form dimers via weak N-H...N hydrogen bonding ( $N...N$  3.054(4) Å).

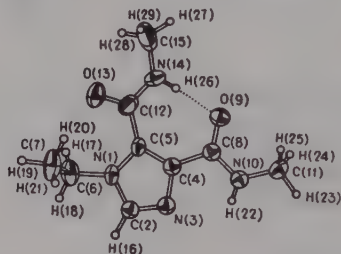


Fig. 1. The  $\text{C}_9\text{H}_{14}\text{N}_4\text{O}_2$  molecule.

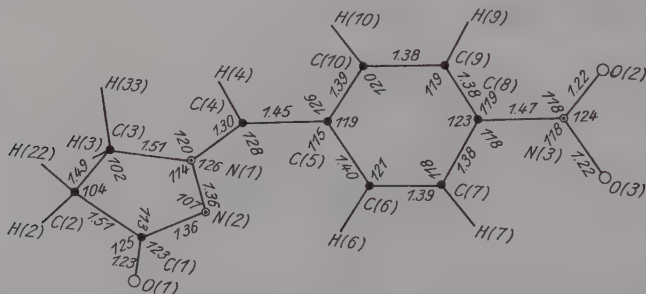
## 1-(4-NITROBENZYLIDENE)-PYRAZOLIDONE-(3)-BETAINE



S. KULPE, I. SEIDEL, G. GEISLER and G. TOMASCHESKI, 1982. Cryst. Res. & Technol., 17, 91-99.

Orthorhombic, Pbca,  $a = 11.025$ ,  $b = 22.995$ ,  $c = 7.677$  Å,  $Z = 8$ . Mo radiation,  $R = 0.047$  for 1516 reflexions.

The phenyl ring atoms are coplanar within experimental error limits but the nitro-group is twisted out of this plane by some  $10.8^\circ$  (Fig. 1). The five-membered ring has an envelope conformation, and the best plane through all five ring atoms forms a dihedral angle of  $19.4^\circ$  with the plane through the phenyl ring. The molecules are packed in two-dimensional layers parallel to the (100) plane. Within these layers there are C-H...O bridges e.g. C(4)-H(4)...O(1') with C(4)...O(1') 3.03 Å, and C(4)-H(4)...O(1')  $157^\circ$ .



2-(CYCLOHEPTA-1,3,5-TRIEN-7-YL)IMIDAZOLIDIN-2-YLIUM TETRAFLUOROBORATE  
 $C_{10}H_{13}BF_4N_2$  (I)

SPIRO(CYCLOHEPTA-1,3,5-TRIEN-7,3'-TETRAHYDROFURAN)-2'-ONE  
 $C_{10}H_{10}O_2$  (II)

W. BAUER, J. DAUB, G. MAAS, M. MICHNA, K.M. RAPP and J.J. STEZOWSKI, 1982. Chem. Ber., 115, 99-118.

I. Orthorhombic,  $P2_12_12_1$ ,  $a = 9.443$ ,  $b = 10.918$ ,  $c = 11.172$  Å,  $D_m = 1.42$ ,  $Z = 4$ . Mo radiation,  $R = 0.056$  for 2690 reflexions.

II. Monoclinic,  $P2_1/n$ ,  $a = 8.810$ ,  $b = 14.894$ ,  $c = 6.331$  Å,  $\beta = 92.21^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.053$  for 832 reflexions.

In I (Fig. 1) the free rotation about the C(7)-C(8) bond results in the imidazolidinium substituent adopting the endo conformation. In II the carbonyl group adopts the exo conformation and the dihedral angle between the two mean ring planes is  $81^\circ$ . In each molecule the cycloheptatriene ring adopts a boat conformation.

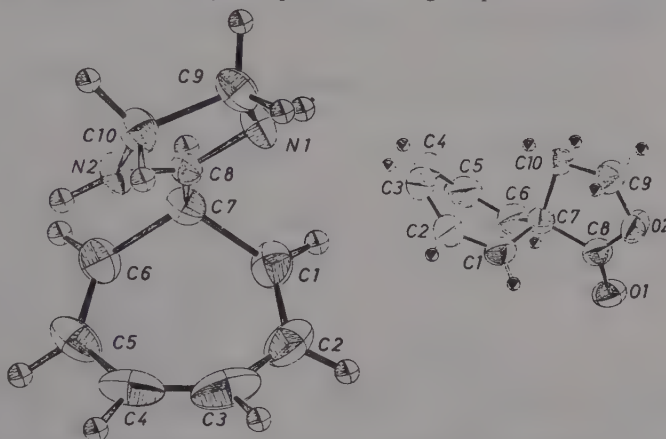


Fig. 1. Perspective view of the  $C_{10}H_{13}N_2^+$  cation (left) and  $C_{10}H_{10}O_2$  (right).

trans-1-OXYL-2,2,5,5-TETRAMETHYL-3,4-DICYANOPYRROLIDINE  
 $C_{10}H_{14}N_3O$

J.F.W. KEANA, K. HIDEG, G.B. BIRRELL, O.H. HANKOVSKY, G. FERGUSON and M. PARVEZ, 1982. Canad. J. Chem., 60, 1439-1447.

Orthorhombic,  $Pbcn$ ,  $a = 19.155$ ,  $b = 7.634$ ,  $c = 15.059$  Å,  $Z = 4$ . Mo radiation,  $R = 0.058$  for 1068 reflexions.

The molecule (Fig. 1) adopts a slightly distorted envelope conformation with C3 at the flap. The N-O distance,  $1.269(4)$  Å, is within the usual range for unconjugated radicals.

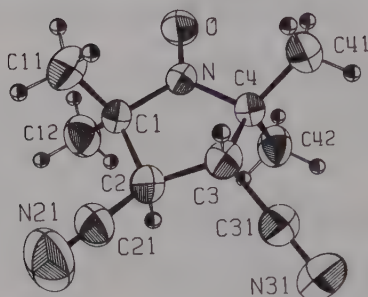


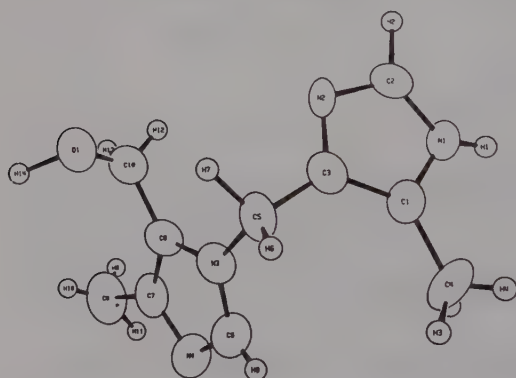
Fig. 1. A view of the  $C_{10}H_{14}N_3O$  molecule.

4-METHYL-1-((5-METHYL-1H-IMIDAZOL-4-YL)METHYL)-1H-IMIDAZOLE-5-METHANOL  
 $C_{10}H_{14}N_3O$

D.F. CHODOSH, B. LAM, C. LABAW and G.R. WELLMAN, 1982. Cryst. Struct. Comm., **11**, 1265-1268.

Orthorhombic,  $Pca2_1$ ,  $a = 23.024$ ,  $b = 4.819$ ,  $c = 9.682$  Å,  $Z = 4$ . Cu radiation,  $R = 0.061$  for 997 reflexions.

Each imidazole ring (Fig. 1) is individually planar; the angle between the planes is  $69.1^\circ$ .



N2-H1	2.13(6)*	N2-C3	1.370(7)
N4-H14	1.63(7)*	N3-C5	1.482(7)
O1-C10	1.407(8)	N3-C6	1.319(7)
O1-H14	1.20(7)	N3-C8	1.379(8)
N1-C1	1.369(8)	N4-C6	1.309(7)
N1-C2	1.343(8)	N4-C7	1.371(8)
N1-H1	0.79(6)	C1-C3	1.388(7)
N2-C2	1.317(8)	C1-C4	1.52(1)

C2-H2	1.19(5)	C7-C8	1.368(7)
C3-C5	1.487(8)	C7-C9	1.508(9)
C4-C3	1.22(9)	C8-C10	1.482(9)
C4-H4	0.89(7)	C9-H9	1.05(7)
C4-H5	0.91(8)	C9-H10	0.86(7)
C5-H6	1.02(8)	C9-H11	1.19(7)
C5-H7	1.15(7)	C10-H12	0.90(7)
C6-H8	0.87(6)	C10-H13	1.01(8)

Fig. 1. The  $C_{10}H_{14}N_4O$  molecule and interatomic distances; values marked with an \* are intermolecular.

2-(2'-AMINO-1'-PROPENYL)-5-DIMETHYLAMINO-4,4-DIMETHYL-4H-IMIDAZOLE  
 $C_{10}H_{18}N_4$  (I)

2-ACETONYLIDENE-5-DIMETHYLAMINO-4,4-DIMETHYL-2,3-DIHYDRO-4H-IMIDAZOLE  
 $C_{10}H_{17}N_3O$  (II)

M. DAHLER, R. PREWO, J.H. BIERI and H. HEIMGARTNER, 1982. Helv. Chim. Acta, **65**, 2302-2312.

I. Tetragonal,  $P4_2/n$ ,  $a = 17.611$ ,  $c = 7.213$  Å,  $Z = 8$ . Mo radiation,  $R = 0.077$  for 3261 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 8.400$ ,  $b = 13.923$ ,  $c = 11.534 \text{ \AA}$ ,  $\beta = 125.35^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.063$  for 4863 reflexions.

Bond lengths for both molecules are in Fig. 1. In the presence of water, I is easily hydrolyzed to II. In I, molecules form chains via N-H...N hydrogen bonds; in II, dimers are formed through N-H...O hydrogen bonds.

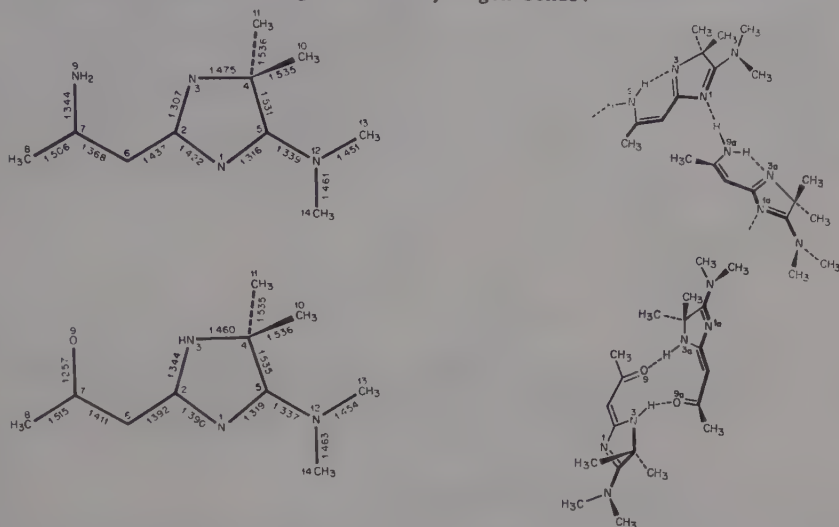


Fig. 1. Bond lengths and hydrogen bonding scheme for  $C_{10}H_{18}N_4$  (I) (top) and  $C_{10}H_{17}N_3O$  (II) (bottom).

# 2-AMINO-5,5-DIMETHYL-1-PYRROLINIUM DIETHYL PHOSPHATE MONOHYDRATE

$C_{10}H_{23}N_2O_4P \cdot H_2O$

$C_6H_{13}N_2^+, C_4H_{10}O_4P^-, H_2O$

E.A.H. GRIFFITH, J.S. RUTHERFORD and B.E. ROBERTSON, 1982. Acta Cryst., B38, 2951-2955.

Monoclinic,  $P2_1/c$ ,  $a = 14.138$ ,  $b = 9.325$ ,  $c = 12.369 \text{ \AA}$ ,  $\beta = 107.71^\circ$ ,  $D_m = 1.21$ ,  $Z = 4$ . Mo radiation,  $R = 0.072$  for 1858 reflexions.

Bond lengths and angles in the amidinium and phosphate ions are generally as expected (Fig. 1). The structure involves four modes of hydrogen bonding between cations and anions, two of which involve water acting as a bridge. The O(ethyl)-P-O-C torsion angles are  $\pm 67.1(6)$  and  $\pm 63.3(6)^\circ$ .



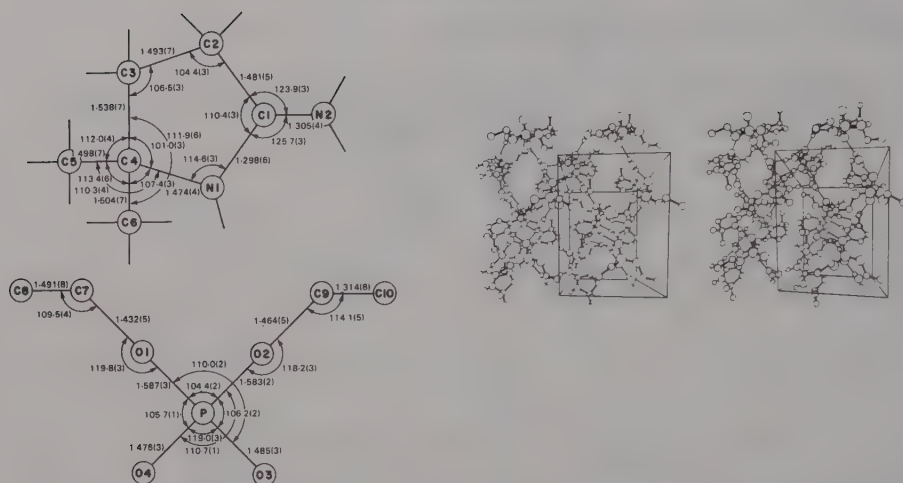
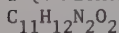


Fig. 1.  $C_6H_{13}N_2^+, C_4H_{10}O_4P^-, H_2O$ : bond lengths and angles in the cation (upper left) and anion (lower left) and a stereoview of the packing of the unit cell, b is horizontal and c vertical (right).

# 1-(4-METHOXYBENZYLIDENE)-PYRAZOLIDONE-(3)-BETAINE



S. KULPE, I. SEIDEL and G. GEISSLER, 1982. Cryst. Res. & Technol., **17**, 1427-1433.

Orthorhombic, Pcam,  $a = 14.512$ ,  $b = 10.516$ ,  $c = 6.812$  Å,  $Z = 4$ . Mo radiation,  $R = 0.039$  for 831 reflexions.

The molecule has crystallographic mirror symmetry with all but a few hydrogen atoms lying in the space group mirror planes at  $Z = 0.25$  and  $Z = 0.75$  (Fig. 1). The interplanar spacing is 3.406 Å. The C(4)-C(5) bond length of 1.440 Å indicates conjugation between the azomethine imine unit with polymethinic character and the methoxybenzylidene substituent with aromatic character. The short C(4)-N(1) bond length of 1.289 Å indicates high positive charge on N(1). Other distances of interest are: N(1)-N(2) 1.368, N(1)-C(3) 1.501, C(1)-O(1) 1.230, C(1)-N(2) 1.342, C(1)-C(2) 1.512 and C(2)-C(3) 1.493 Å. There are C-H...O hydrogen bonds between molecules in the same plane with C(4)...O(1') 3.13 Å and C(4)-H(4)...O(1') 174°.

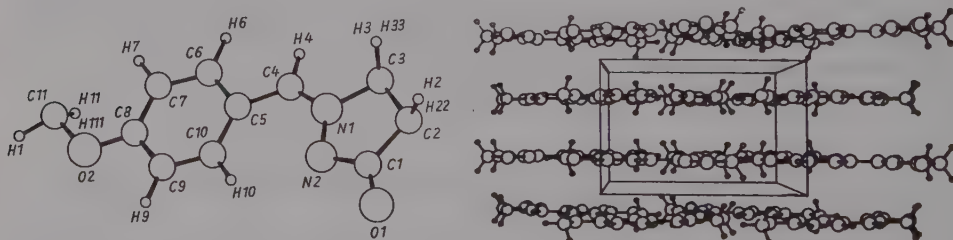
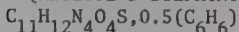


Fig. 1. Atomic numbering scheme in  $C_{11}H_{12}N_2O_2$ , and molecular packing diagram viewed down the b axis.

# 1-(MESITYL-2-SULFONYL)-3-NITRO-1,2,4-TRIAZOLE BENZENE SOLVATE



R. KURODA, M.R. SANDERSON, S. NEIDLE and C.B. REESE, 1982. J. Chem. Soc. Perkin II, 617-620.

Triclinic,  $P\bar{1}$ ,  $a = 8.001$ ,  $b = 8.140$ ,  $c = 12.892$  Å,  $\alpha = 98.22$ ,  $\beta = 105.49$ ,  $\gamma = 102.12^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.074$  for 2926 reflexions.

The N(1)-N(2) bond length (1.360) (Fig. 1) shows considerable double bond character as do the ring C-N bonds which range from 1.309 to 1.341 Å. The C(3)-N(11) bond (1.447 Å) to the nitro group shows little double bond character and the N(11)-ON(1) and N(11)-ON(2) bonds are at the extreme for N-O bonds at 1.219 and 1.229 Å respectively. The mesitylene ring is planar and staggered with respect to the triazole ring, the angle between the normals to the two rings being  $80.4^\circ$ . There is partial stacking between the triazole ring and the benzene solvent molecule, but the benzene ring is somewhat inclined to the plane of the triazole ring.

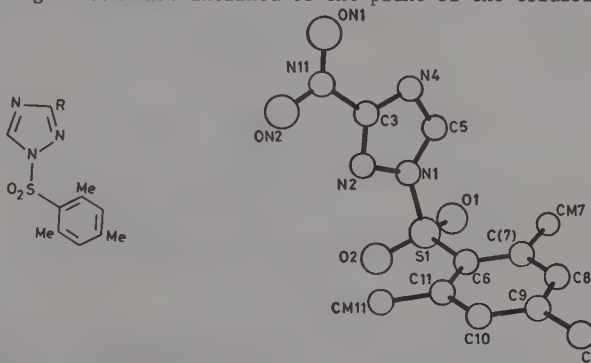


Fig. 1. Molecular skeleton ( $R = NO_2$ ) and structure of  $C_{11}H_{12}N_4O_4S \cdot 0.5(C_6H_6)$ .

# 1-PHENYL-3,5-DIMETHYLPYRAZOLIUM NITRATE

$C_{11}H_{13}N_3O_3$

$C_{11}H_{13}N_2^+ \cdot NO_3^-$

J. ZUCKERMAN-SCHPECTOR, E.E. CASTELLANO, A.C. MASSABNI and A.D. PINTO, 1982. Canad. J. Chem., 60, 97-99.

Monoclinic,  $P2_1/n$ ,  $a = 7.916$ ,  $b = 10.934$ ,  $c = 13.638$  Å,  $\beta = 97.37^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.055$  for 1099 reflexions.

Bond lengths and numbering scheme are shown in Fig. 1 along with possible resonance forms for the five-membered ring. The cation and anion are linked by an N-H...O hydrogen bond ( $N(2) \cdots O(1)$  2.66 Å). The dihedral angle between the phenyl and pyrazolium ring planes is  $48.2^\circ$ .

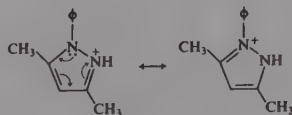
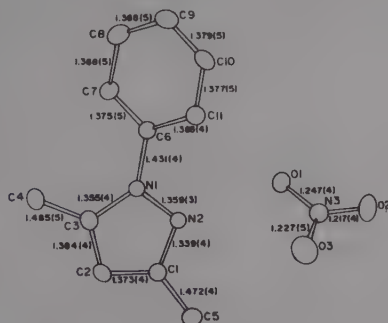


Fig. 1. Bond distances and possible resonance forms for the five-membered ring in  $C_{11}H_{13}N_3O_3$ .

## 1,4-DIMETHYL-5-PHENYL-5-VINYL-2-TETRAZOLINE

 $C_{11}H_{14}N_4$ 

M.M. GRANGER, L. TOUPET and J.Y. LE MAROUILLE, 1982. Cryst. Struct. Comm., **11**, 75-78.

Triclinic,  $P\bar{1}$ ,  $a = 7.472$ ,  $b = 8.688$ ,  $c = 9.159$  Å,  $\alpha = 93.54$ ,  $\beta = 96.43$ ,  $\gamma = 110.81^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.042$  for 1436 reflexions.

Molecular dimensions are shown in Fig. 1. The five-membered ring has an envelope conformation with a  $29.3^\circ$  flat (at C7).

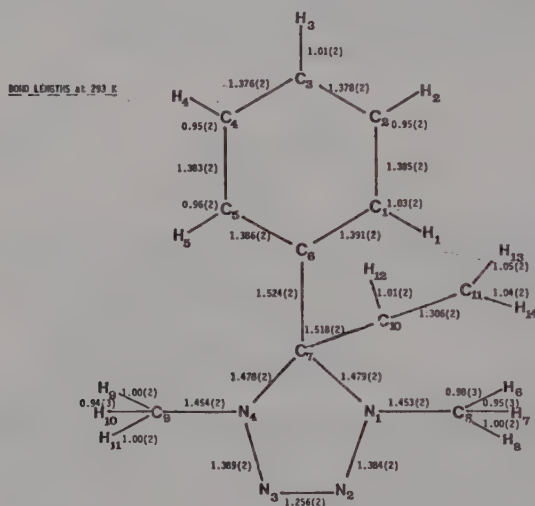


Fig. 1. Bond lengths in  $C_{11}H_{14}N_4$ .

## 2-[5-(2-PYRIDYL)-1H-1,2,4-TRIAZOL-3-YL]PYRIDINIUM PERCHLORATE

 $C_{12}H_{10}ClN_5O_4$  $C_{12}H_{10}N_5^+, ClO_4^-$ 

R. PRINS, P.J.M.W.L. BIRKER and G.C. VERSCHOOR, 1982. Acta Cryst., **B38**, 2934-2935.

Monoclinic,  $C2/c$ ,  $a = 32.889$ ,  $b = 5.311$ ,  $c = 15.712$  Å,  $\beta = 104.95^\circ$ ,  $D_m = 1.62$ ,  $Z = 8$ . Mo radiation,  $R = 0.042$  for 1286 reflexions.

The crystal structure consists of hydrogen-bonded ion-pairs, with perchlorate to pyridinium (N-H) hydrogen bonds ( $N...O$  2.795(5) Å) (Fig. 1). There are additional hydrogen bonds between the triazole (N-H) group and a pyridine N atom in adjacent cations ( $N...N$  3.006(5) Å). The  $C_{12}H_{10}N_5^+$  ion is nearly planar.

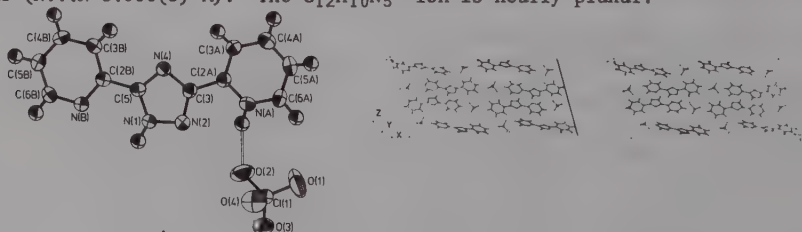
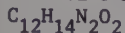


Fig. 1.  $C_{12}H_{10}N_5^+, ClO_4^-$ : views of the structure and of the crystal packing.

## 5-ETHYL-3-METHYL-5-PHENYLHYDANTOIN



M. VAN MEERSSCHE, J.P. DECLERCQ, G. GERMAIN, R. BOUCHÉ and M. DRAGUET-BRUGHMANS, 1982. Bull. Soc. Chim. Belg., 91, 199-204.

Orthorhombic, Pcab,  $a = 27.493$ ,  $b = 8.516$ ,  $c = 10.037$  Å,  $Z = 8$ . Mo radiation,  $R = 0.049$  for 1060 reflexions.

The molecular dimensions are shown in Fig. 1 and are in accord with anticipated values.

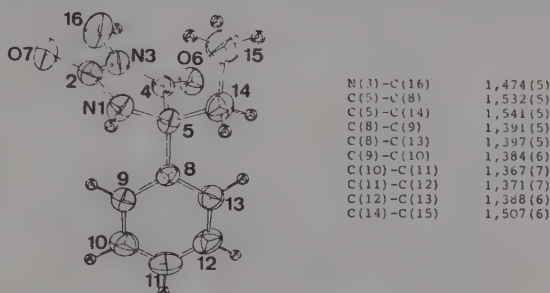
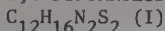


Fig. 1. The  $C_{12}H_{14}N_2O_2$  molecule and bond lengths.

## 3,4-DIPYRROLIDINO-3-CYCLOBUTENE-1,2-DITHIONE



## 2,4-DIPYRROLIDINO-3-THIOXO-CYCLOBUTENYLIUM-1-THIOLATE



R. MATTES, D. ALTMEPPEN, G. JOHANN, M. SCHULTE-COERNE and H. WEBER, 1982. Monatsh. Chem., 113, 191-196.

I. Orthorhombic, Pbcn,  $a = 18.436$ ,  $b = 8.762$ ,  $c = 7.376$  Å,  $Z = 4$ . Mo radiation,  $R = 0.065$  for 1017 reflexions.

II. Orthorhombic, Pbcn,  $a = 18.974$ ,  $b = 8.801$ ,  $c = 7.398$  Å,  $Z = 4$ . Mo radiation,  $R = 0.074$  for 613 reflexions.

Both molecules contain the  $C_4N_2S_2$  group which is approximately planar (Fig. 1), and both molecules have crystallographic symmetry, twofold in I and inversion in II. The mean C-C bond distances in the four-membered ring systems are 1.461 Å in I and 1.438 Å in II. The C-S and C-N distances are 1.645 and 1.313 Å in I, and 1.651 and 1.329 Å in II. The two endocyclic C-N distances are 1.492 and 1.484 Å in I, and 1.460 and 1.474 Å in II and the C-C distances 1.511, 1.522 and 1.529 Å in I and 1.521, 1.505 and 1.511 Å in II.

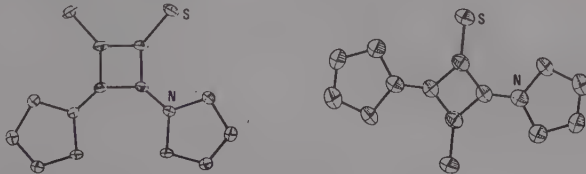


Fig. 1. Molecular structures of the two isomers of  $C_{12}H_{16}N_2S_2$ , I (left) and II (right).

N-(3,3-DIMETHYL-5,5-BIS(TRIFLUOROMETHYL)-1-PYRAZOLINIO)PENTAFLUOROANILIDE  
 $C_{13}H_8F_{11}N_3$

A. GIEREN and V. LAMM, 1982. Z. Naturforsch., **37b**, 1606-1611.

Monoclinic,  $P2_1/n$ ,  $a = 8.761$ ,  $b = 9.773$ ,  $c = 18.727$  Å,  $\beta = 100.86^\circ$ ,  $D_m = 1.73$ ,  $Z = 4$ .  
 Cu radiation,  $R = 0.060$  for 2030 reflexions.

The N-N distances in the mesoionic  $N(3)^- - N(1)^+ = N(2)$  moiety are asymmetric (Fig. 1). The almost planar pyrazolinium ring system and the plane of the pentafluorophenyl group have a dihedral angle of  $61.0(3)^\circ$  caused mainly by a torsion around the N-C bond to the phenyl ring.

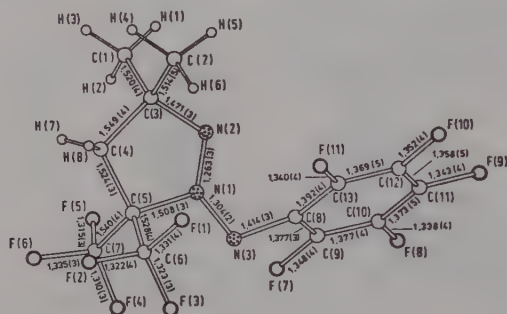


Fig. 1. Bond lengths (Å) and e.s.d.'s in  $C_{13}H_8F_{11}N_3$ .

1-(2,4-DIBROMOPHENYLAMINO)-5-PHENYL-TETRAZOLE  
 $C_{13}H_9Br_2N_5$

Z. DAUTER, S.A. CHAUDHURY and M.A. HAMID, 1982. Cryst. Struct. Comm., **11**, 999-1004.

Monoclinic,  $P2_1/c$ ,  $a = 8.413$ ,  $b = 21.495$ ,  $c = 8.003$  Å,  $\beta = 92.84^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.068$  for 1495 reflexions.

The analysis establishes the position of the dibromophenyl group and that compound is a tetrazole (Fig. 1).

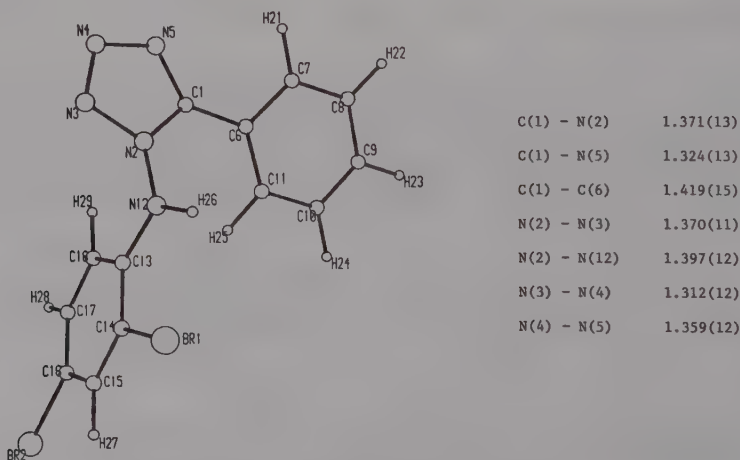


Fig. 1. The  $C_{13}H_9Br_2N_5$  molecule and some bond lengths.

1-(4-CHLOROPHENOXY)-3,3-DIMETHYL-1-(1,2,4-TRIAZOL-1-YL)-2-BUTANONE  
 $C_{14}H_{16}ClN_3O_2$

I.W. NOWELL, P.E. WALKER and N.H. ANDERSON, 1982. Acta Cryst., B38, 1857-1859.

Monoclinic,  $P2_1/n$ ,  $a = 8.076$ ,  $b = 20.317$ ,  $c = 9.307$  Å,  $\beta = 97.43^\circ$ ,  $D_m = 1.31$ ,  $Z = 4$ .  
 Mo radiation,  $R = 0.058$  for 1023 reflexions.

The planar triazolyl ring shows significant delocalisation (e.g. C(1)-N(1) 1.332, C(2)-N(3) 1.336, C(1)-N(3) 1.319, C(2)-N(2) 1.311, N(1)-N(2) 1.348 Å) and is inclined at  $73.1(9)^\circ$  to the aromatic ring and at  $61.8(9)^\circ$  to the C(31),C(4),O(1),C(5) plane leading to short intramolecular N...H contacts (Fig. 1) and to distortion of the O(2)-C(71)-C(72) angle ( $124.2(7)^\circ$ ). The exocyclic angles at N(1) are  $119.5(6)^\circ$  (C-N-N) and  $130.7(6)^\circ$  (C-N-C).

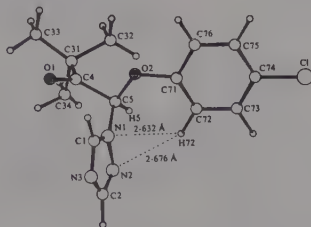


Fig. 1. The conformation of the  $C_{14}H_{16}ClN_3O_2$  molecule.

1-(4-CHLOROPHENOXY)-1-(1,2,4-TRIAZOLYL)-3,3-DIMETHYLBUTAN-2-OL  
 $C_{14}H_{18}ClN_3O_2$

T. SPITZER, J. KOPF and G. NICKLESS, 1982. Cryst. Struct. Comm., 11, 315-320.

Monoclinic,  $P2_1/c$ ,  $a = 17.298$ ,  $b = 5.864$ ,  $c = 16.608$  Å,  $\beta = 117.28^\circ$ ,  $Z = 4$ . Mo  
 radiation,  $R = 0.084$  for 1610 reflexions.

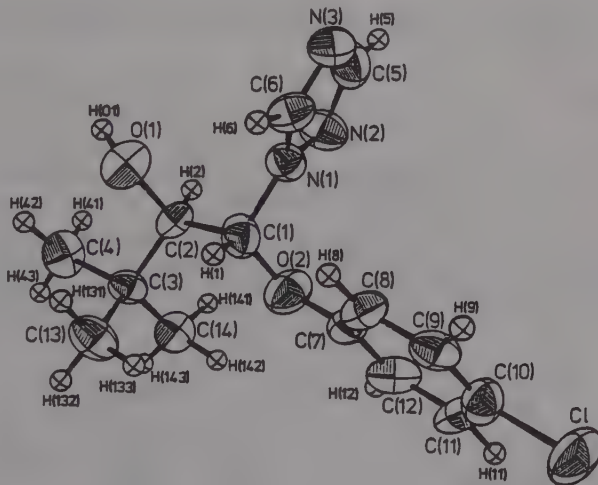


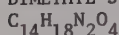
Fig. 1. A view of the  $C_{14}H_{18}ClN_3O_2$  molecule.

The molecular structure is shown in Fig. 1. The angle between the best mean planes through C(7) to C(12) and the triazole ring N(1) to C(6) is  $95.2^\circ$ . The two chiral C-atoms C(1) and C(2) both have R,R (S,S) configuration. The R,R (S,S) configuration corresponds with the  $^1H$ -NMR spectrum in solution which gives evidence for an almost trans configuration of the H-atoms on C(1) and C(2) (solvent:  $CDCl_3$ ,



coupling constant 3.6 Hz). Both diastereomers can be distinguished by their  $^{13}\text{C}$ -NMR-spectra, but the differences in chemical shifts are below 1 ppm for all C-atoms.

# DIMETHYL 5-METHYL-2-PHENYL-3,4-PYRAZOLIDINEDICARBOXYLATE



L. TOUPET, Y. DÉLUGEARD and J.C. MESSEGER, 1982. *Acta Cryst.*, **B38**, 1377-1380.

Monoclinic,  $P2_1/c$ ,  $a = 10.03$ ,  $b = 14.46$ ,  $c = 20.60$  Å,  $\beta = 100.24^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.054$  for 2041 reflexions.

The two independent molecules (Fig. 1) in the asymmetric unit have very similar bond lengths and angles and are linked into dimers by hydrogen bonds.

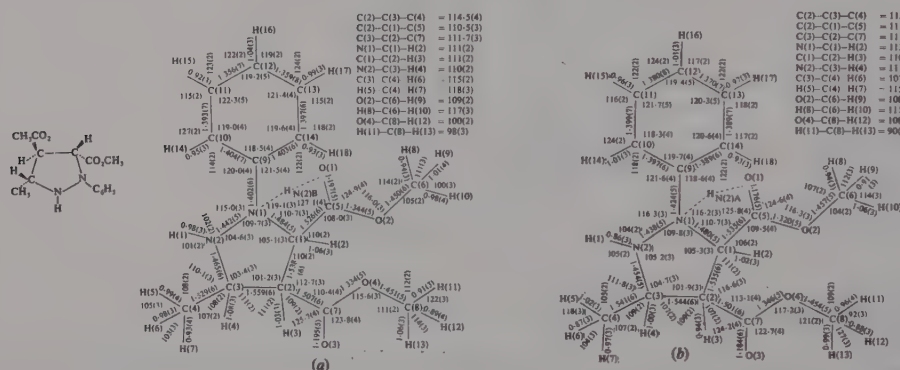
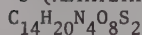


Fig. 1.  $\text{C}_{14}\text{H}_{18}\text{N}_2\text{O}_4$ : bond lengths and angles in molecule A and molecule B.

# 3,4,5-TRIS(METHOXYCARBONYL)-4-(N'-(METHOXYCARBONYL)(METHYLTHIO)METHYLENE)HYDRAZINO)-5-(METHYLTHIO)-2-PYRAZOLINE



T. KAMPCHEN, W. MASSA, W. OVERHEU, R. SCHMIDT and G. SEITZ, 1982. *Chem. Ber.*, **115**, 683-694.

Triclinic,  $P\bar{1}$ ,  $a = 16.802$ ,  $b = 17.233$ ,  $c = 7.378$  Å,  $\alpha = 90.41$ ,  $\beta = 93.06$ ,  $\gamma = 73.40^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.046$  for 4705 reflexions.

The pyrazolin ring (Fig. 1) adopts an envelope conformation and the ester group at C(3) is nearly coplanar with the N(2)-C(3) double bond but the orientation is cis and trans in the five independent molecules ( $\text{N}(2)\text{C}(3)-\text{C}(6)\text{O}(1)$  17.7,  $-163.1^\circ$ ). The  $\text{N}(4)-\text{C}(8)$  double bond (1.285(4) Å) has the Z-configuration ( $\text{N}(4)\text{C}(8)-\text{C}(10)\text{O}(3)$  159.9,  $170.0^\circ$ ).

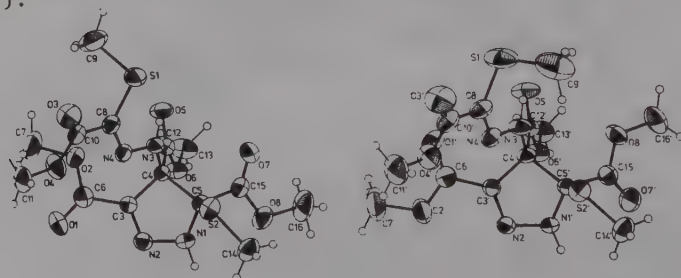


Fig. 1. The two independent molecules of  $\text{C}_{14}\text{H}_{20}\text{N}_4\text{O}_8\text{S}_2$ .

(S)-(+)- $\alpha$ -(p-BROMOPHENYL)ETHYLAMMONIUM 1-METHOXYAZIRIDINE-2,2-DICARBOXYLIC ACID *cis*-ETHYL ESTER

$C_{15}H_{21}BrN_2O_5$

$C_8H_{11}BrN^+, C_7H_{10}NO_5^-$

V.F. RUDCHENKO, O.A. D'YACHENKO, A.B. ZOLOTI, L.O. ATOVMYAN, I.I. CHERVIN and R.G. KOSTYANOVSKY, 1982. *Tetrahedron*, **38**, 961-975.

Orthorhombic,  $P2_12_12_1$ ,  $a = 25.121$ ,  $b = 10.512$ ,  $c = 7.062$  Å,  $Z = 4$ . Cu radiation,  $R = 0.050$  for 1309 reflexions.

The analysis determined the absolute configuration of the anion as shown in Fig. 1 with *trans*-orientation of  $CO_2^-$  and MeO groups.

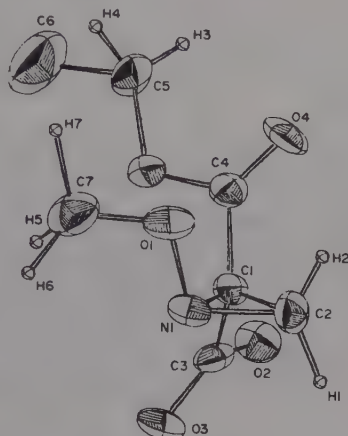


Fig. 1. A view of the methoxy-aziridine anion.

4-(N,N-DIMETHYL-N-ETHYL-AMMONIUM)-2,3-DIMETHYL-1-PHENYL-3-PYRAZOLIN-5-ONE IODIDE

$C_{15}H_{22}IN_3O$

Gy. ARGAY, B. RIBAR, V. DIVJAKOVIC, D. ZIVANOV-STAKIC, S. VLADIMIROV, Lj. MANOJLVOIC-MUIR, 1982. *Cryst. Struct. Comm.*, 1193-1197.

Tetragonal,  $P4_1$ ,  $a = 10.340$ ,  $c = 15.663$  Å,  $D_m = 1.55$ ,  $Z = 4$ . Mo radiation,  $R = 0.077$  for 2062 reflexions.

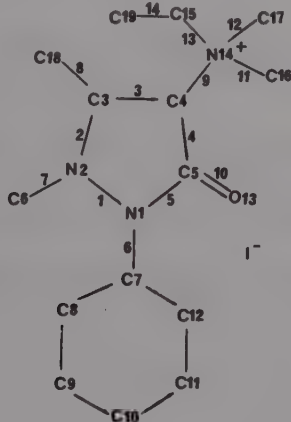
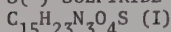


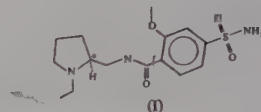
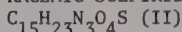
Fig. 1. A schematic view of  $C_{15}H_{22}IN_3O$ .

The pyrazoline ring (Fig. 1) is almost planar. The greatest endocyclic torsion angle is  $2.3^\circ$ . The best plane of the pyrazoline ring (max. dev.  $0.01 \text{ \AA}$ ) makes a dihedral angle  $62.6^\circ$  with that of the phenyl ring. The ring nitrogens have a planar geometry.

S(-)-SULPIRIDE



RACEMIC SULPIRIDE



L.Y.Y. MA, N. CAMERMAN and A. CAMERMAN, 1982. *Acta Cryst.*, B38, 2861-2865.

I. Orthorhombic,  $P2_12_12_1$ ,  $a = 12.037$ ,  $b = 24.163$ ,  $c = 11.536 \text{ \AA}$ ,  $Z = 8$ . Cu radiation,  $R = 0.0653$  for 1401 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 9.067$ ,  $b = 9.372$ ,  $c = 11.175 \text{ \AA}$ ,  $\alpha = 65.66$ ,  $\beta = 79.83$ ,  $\gamma = 76.79^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.0738$  for 1536 reflexions.

In the crystal structures the conformations of the racemate (II) and one of the two independent molecules of S(-)-sulpiride (I) are very similar (Fig. 1). Both molecules have extended rather flattened conformations and differ only in orientations of freely rotatable substituent groups at both ends. The second enantiomeric molecule in I differs greatly in the conformational relationship of the five-membered, heterocyclic ring to the rest of the molecule. The phenyl ring and amide linkage in all three molecules are roughly planar; the five-membered ring is not, and exhibits conformational flexibility. In both structures the molecules are linked in the crystal by hydrogen bonds. For another study of II see following Report.

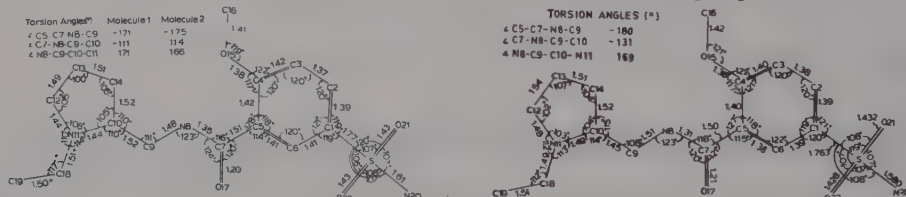
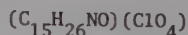
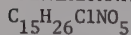
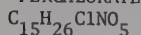


Fig. 1.  $C_{15}H_{23}N_3O_4S$ : bond lengths and angles in S(-)-sulpiride (I) (averaged values) and in racemic sulpiride (II).

N-(7,7-DIMETHYLBICYCLO[3.2.0]HEPTAN-6-YLIDENE)-2-METHOXYMETHYLPYRROLIDINIUM



C. HOUGE, A.M. FRISQUE-HESBAIN, A. MOCKEL, L. GHOSZ, J.P. DECLERCQ, G. GERMAIN and M. VAN MEERSSCHE, 1982. *J. Am. Chem. Soc.*, 104, 2920-2921.

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.178$ ,  $b = 14.035$ ,  $c = 17.331 \text{ \AA}$ ,  $Z = 4$ . Mo radiation,  $R = 0.070$  for 1111 reflexions (absolute configuration).

The configuration of the ring fusion was found to be (1R,5S). Molecular dimensions are available from the Cambridge Crystallographic Data Centre.

2,2',3,3',4,4',5,5'-OCTAKIS(TRIFLUOROMETHYLTHIO)-1,1'-BIPYRROLE

$C_{16}F_{24}N_2S_8$

$(C_8F_{12}NS_4)_2$

M.R.C. GERSTENBERGER, A. HAAS, B. KIRSTE, C. KRUGER and H. KURRECK, 1982. Chem. Ber., 115, 2540-2547.

Monoclinic,  $C2/c$ ,  $Z = 4$ .  $R = 0.10$ .

The molecule (Fig. 1) has  $C_2$  symmetry with  $N(1)-N(2)$  1.38,  $S-CF_3$  1.75,  $C-F$  1.33 Å. The dihedral angle between the planes of the pyrrole rings is  $92.8^\circ$ .

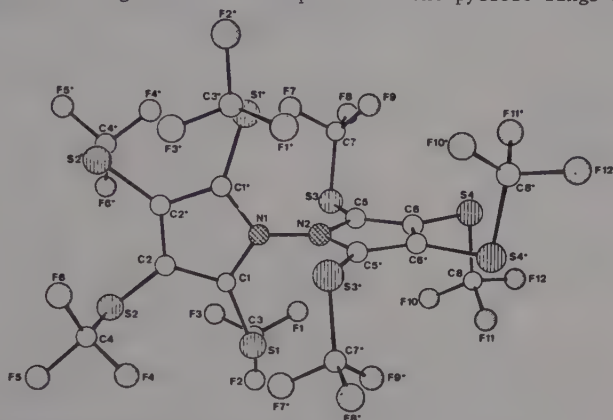


Fig. 1. The structure of  $(C_8F_{12}NS_4)_2$ .

1-PHENYL-3-METHYL-4-(4'-CHLOROPHENYLAZO)-PYRAZOL-5-ONE

$C_{16}H_{13}ClN_4O$

B. GOLINSKI, G. RECK and L. KUTSCHABSKY, 1982. Z. Krist., 158, 271-278.

Monoclinic,  $P2_1/n$ ,  $a = 13.94$ ,  $b = 22.81$ ,  $c = 4.80$  Å,  $\beta = 92.5^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.16$  for 1012 reflexions.

The molecule occurs as the hydrazone tautomer and is packed in columns separated by van der Waals distances.

1-(1,1,3,3-TETRAMETHYL)BUTYL-4-PHENYL-1,2,4-TRIAZOLINE-3,5-DIONE

$C_{16}H_{23}N_3O_2$

R.J. BAKER, J.W. TIMBERLAKE, J.T. ALENDER, R.J. MAJESTE and L.M. TREFONAS, 1982. Cryst. Struct. Comm., 11, 763-768.

Monoclinic,  $P2_1/c$ ,  $a = 6.525$ ,  $b = 19.654$ ,  $c = 15.501$  Å,  $\beta = 126.13^\circ$ ,  $D_m = 1.20$ ,  $Z = 4$ . Cu radiation,  $R = 0.049$  for 1501 reflexions.

In this molecule (Fig. 1) the phenyl ring plane is inclined at  $43.6^\circ$  to the triazoline ring plane.



Fig. 1. Molecular geometry and a stereoview of  $C_{16}H_{23}N_3O_2$ .

**N,N-DIETHYL-1-HYDROXY-4-METHOXY-2-METHYL-3-PHENYL-2-AZETIDINECARBOXAMIDE**  
 $C_{16}H_{24}N_2O_3$

M.L.M. PENNING, D.N. REINHOUDT, S. HARKEMA and G.J. VAN HUMMEL, 1982. *J. Org. Chem.*, **47**, 4419-4425.

Monoclinic,  $P2_1/c$ ,  $a = 17.647$ ,  $b = 14.358$ ,  $c = 13.943$  Å,  $\beta = 111.99^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.137$  for 2191 reflexions.

The analysis reveals that the compound has the structure shown in Fig. 1.

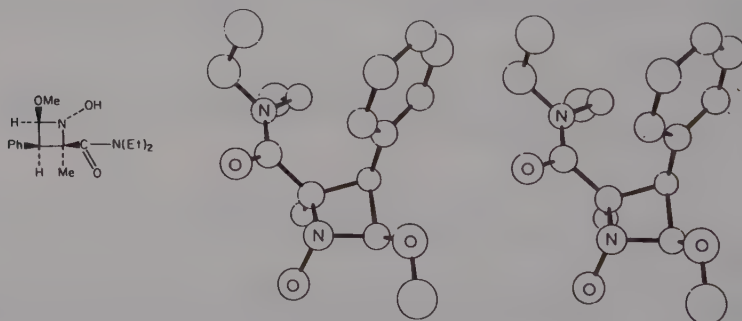


Fig. 1. The  $C_{16}H_{24}N_2O_3$  molecule and a stereoview.

**2,3-DIMETHYL-4-DIMETHYLAMINO-1-PHENYL-1,2-DIAZOL-5-ONE TRISTHIIOUREA**  
 $C_{16}H_{29}N_9OS_3$

$C_{13}H_{17}N_3O_3CS(NH_2)_2$

H. WIEDENFELD, A. KIRFEL and G. KOOP, 1982. *Arch. Pharm.*, **315**, 633-641.

Triclinic,  $P\bar{1}$ ,  $a = 9.378$ ,  $b = 11.724$ ,  $c = 11.880$  Å,  $\alpha = 69.55^\circ$ ,  $\beta = 83.55^\circ$ ,  $\gamma = 75.33^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.045$  for 4356 reflexions.

The asymmetric unit contains one aminophenazone and three thiourea molecules, the latter linked in a three-dimensional lattice of hydrogen bonds. The aminophen-

azone molecules (Fig. 1) lie in the hollow formed within this lattice.

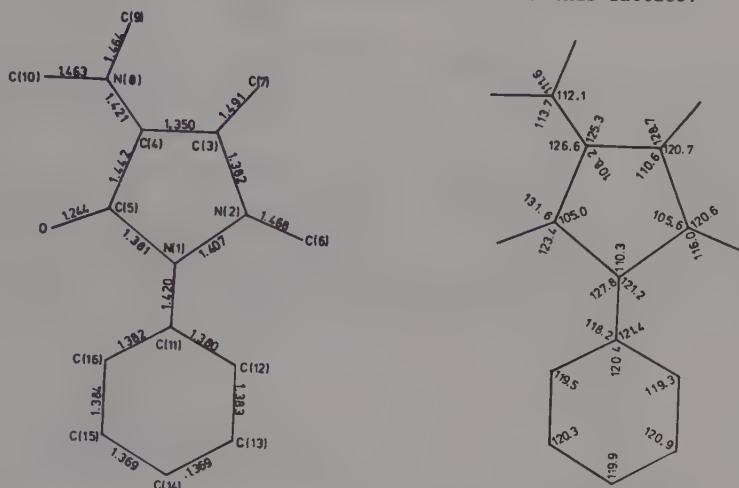
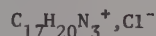
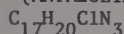


Fig. 1. Bond lengths and angles in the  $C_{13}H_{17}N_3O$  molecule.

4,5-DIHYDRO-N-PHENYL-N-(PHENYLMETHYL)-1H-IMIDAZOLE-2-METHANAMINE HYDROCHLORIDE  
(ANTAZOLINE HYDROCHLORIDE)



V. BERTOLASI, P.A. BOREA and G. GILLI, 1982. *Acta Cryst.*, B38, 2522-2525.

Monoclinic,  $I2/c$ ,  $a = 25.819$ ,  $b = 5.917$ ,  $c = 21.549$  Å,  $\beta = 104.30^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.041$  for 2086 reflexions.

The molecule (Fig. 1) is protonated at one N of the imidazoline ring, with the result that the C(15)-N(2) and C(15)-N(3) distances, 1.294(2) and 1.308(2) Å respectively, are intermediate between those for single and double bonds. Other bond distances and angles are generally normal, except the distance N(1)-C(1) of 1.398(2) Å which is longer than expected. The present analysis, taken with previous analyses, seems to substantiate the idea that a distance of 6.00-6.40 Å between the amino N and the centre of gravity of one unsaturated ring is the main requirement to be fulfilled for the molecule to display antihistaminic activity.

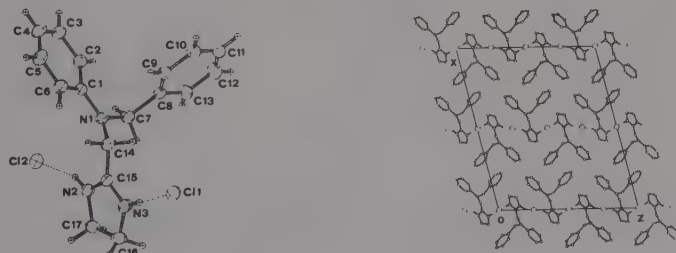


Fig. 1.  $C_{17}H_{20}N_3^+, Cl^-$ : molecular structure and molecular packing in the crystal.



1-[ANTHRYL-(9)-METHYLENE]-PYRAZOLIDONE-(3)-BETAINE  
 $C_{18}H_{14}N_2O$

S. KULPE, I. SEIDEL and G. GEISSLER, 1982. *Cryst. Res. & Technol.*, **17**, 1419-1425.

Orthorhombic,  $Pbca$ ,  $a = 19.541$ ,  $b = 14.800$ ,  $c = 9.572$  Å,  $Z = 8$ . Mo radiation,  $R = 0.077$  for 1177 reflexions.

The conjugation of the azomethine imine unit with the anthryl unit is very small (Fig. 1) as indicated by the bond length 1.480 Å for C(4)-C(5), and by the dihedral angle of  $65.4^\circ$  between the anthryl substituent and the five-membered ring. A positive charge at N(1) is assumed in view of the short (1.288 Å) length of N(1)-C(4). The five-membered ring has an envelope conformation with bond lengths: C(1)-O(1) 1.241, C(1)-C(2) 1.514, C(2)-C(3) 1.496, C(3)-N(1) 1.488, N(1)-N(2) 1.361 and C(1)-N(2) 1.341 Å. The molecules are linked by C(4)-H(4)...N(2') hydrogen bonds with C(4)...N(2) 3.30 Å and C(4)-H(4)...N(2')  $133^\circ$ .

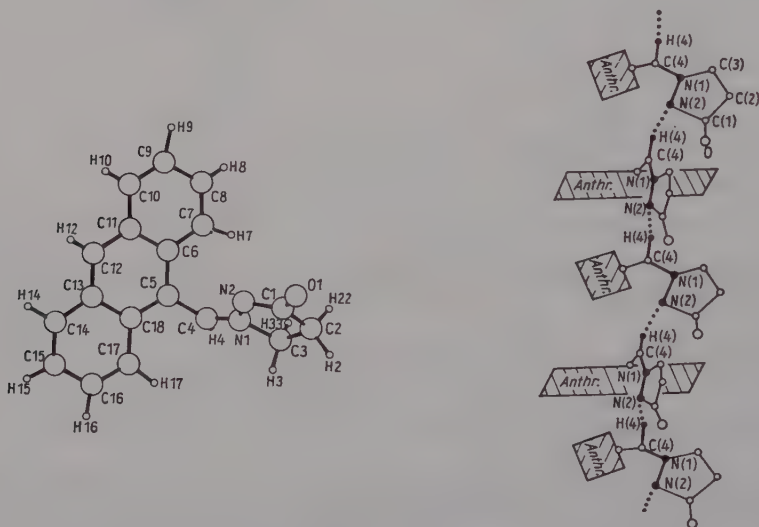


Fig. 1. Molecular structure (left), and intermolecular contacts (right) in  $C_{18}H_{14}N_2O$ .

LACTAM-N-(4-NITROPHENYL)-2-OXO-3-CHLORO-4-PHENYL-4'-CARBETHOXY-AZETIDINE  
 $C_{18}H_{15}ClN_2O_5$

G.D. NIGAM, S. GHOSH and G.B. MITRA, 1982. *Cryst. Struct. Comm.*, **11**, 1677-1682.

Monoclinic,  $P2_1/a$ ,  $a = 10.559$ ,  $b = 15.410$ ,  $c = 11.313$  Å,  $\beta = 109.62^\circ$ ,  $D_m = 1.43$ ,  $Z = 4$ . Cu radiation,  $R = 0.050$  for 2026 reflexions.

Bond lengths are shown in Fig. 1. The four-membered ring is planar, the nitrophenyl and phenyl ring planes are inclined at  $15.5$  and  $105.4^\circ$  respectively.

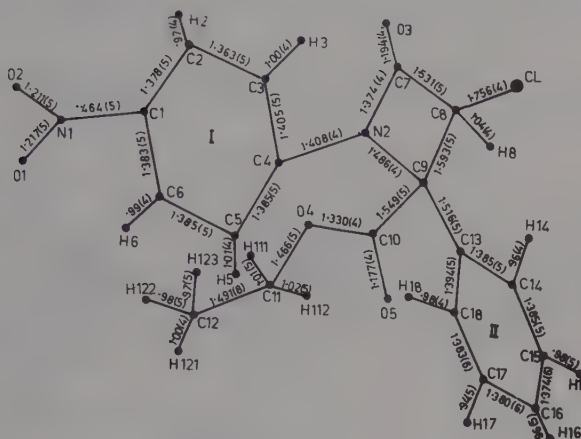


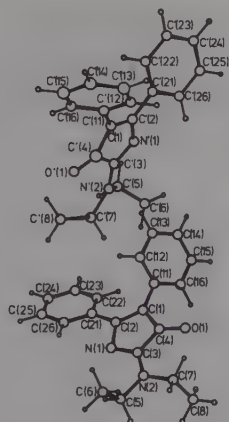
Fig. 1. Bond lengths in  $C_{18}H_{15}ClN_2O_5$ .

2-DIETHYLAMINO-4,5-DIPHENYL-3H-PYRROL-3-ONE  
C<sub>20</sub>H<sub>20</sub>N<sub>2</sub>O

H. PREUT, T. EICHER, G. FRANKE and C. KRÜGER, 1982. *Acta Cryst.*, B38, 1368-1370.

Monoclinic,  $P2_1/a$ ,  $a = 13.195$ ,  $b = 17.389$ ,  $c = 15.542$  Å,  $\beta = 108.07^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.065$  for 2979 reflexions.

The asymmetric unit contains two molecules (Fig. 1) which have significant differences in bond lengths and angles and which are deformed differently by packing forces.



	Molecule 1	Molecule 2
C(1)–C(2)	1.385 (5)	1.378 (5)
C(2)–N(1)	1.426 (5)	1.398 (5)
N(1)–C(3)	1.357 (5)	1.322 (5)
C(3)–C(4)	1.505 (5)	1.539 (6)
C(4)–C(1)	1.423 (6)	1.439 (6)
C(1)–C(11)	1.477 (5)	1.477 (5)
C(2)–C(21)	1.477 (5)	1.479 (4)
C(3)–N(2)	1.328 (4)	1.321 (6)
N(2)–C(5)	1.496 (6)	1.501 (6)
C(5)–C(6)	1.494 (7)	1.454 (7)
N(2)–C(7)	1.469 (4)	•
C(7)–C(8)	1.514 (7)	•
C(4)–O(1)	1.236 (4)	1.225 (5)

Fig. 1.  $\text{C}_{20}\text{H}_{20}\text{N}_2\text{O}$ : a perspective view and bond lengths of the two molecules. Atoms C(7) and C(8) of molecule 2 are disordered.

N-[(2RS,3RS)-1-BENZYL-2-METHYL-3-PYRROLIDINYL]-5-CHLORO-2-METHOXY-4-METHYLAMINO-BENZAMIDE  
C<sub>21</sub>H<sub>26</sub>ClN<sub>3</sub>O<sub>2</sub>

T. FURUYA, S. IWANAMI, A. TAKENAKA and Y. SASADA, 1982. Bull. Chem. Soc. Jpn., 55, 2321-2326.

Monoclinic,  $P2_1/c$ ,  $a = 11.694$ ,  $b = 12.117$ ,  $c = 14.482$  Å,  $\beta = 100.01^\circ$ ,  $D_m = 1.27$ ,  $Z = 4$ . Mo radiation,  $R = 0.082$  for 2752 reflexions.

There is an intramolecular N-H...O hydrogen bond found in the structure as shown in Fig. 1. The pyrrolidine ring has a conformation intermediate between envelope and half-chair.

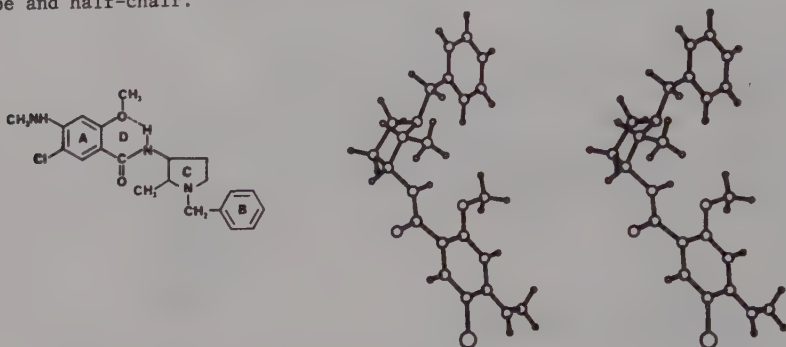


Fig. 1. The  $C_{21}H_{26}ClN_3O_2$  molecule and a stereoview.

DIETHYL 2,4-DIPHENYL-1-PYRROLINE-5,5-DICARBOXYLATE  
 $C_{22}H_{23}NO_4$

F. THÉOBALD, M. BIROUK and J.-F. ROBERT, 1982. *Acta Cryst.*, **B38**, 1764-1768.

Triclinic,  $P\bar{1}$ ,  $a = 8.654$ ,  $b = 14.012$ ,  $c = 8.472$  Å,  $\alpha = 98.97$ ,  $\beta = 102.89$ ,  $\gamma = 102.35^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.064$  for 3998 reflexions.

In the molecule (Fig. 1) the mean plane of the pyrroline ring makes angles with the phenyl groups of  $8^\circ$  (C11-C16) and  $65^\circ$  (C5-C10). C(3) and C(5) are  $-0.292(5)$  and  $0.414(4)$  Å respectively from the C(4), N, C(1), C(2), C(11) plane, with C(5) equatorial. In the ester groups C(18) and C(19) are  $0.053(6)$  and  $-0.366(6)$  Å from the C(4), C(17), O(1), O(2) plane; C(21) and C(22) are  $0.030(5)$  and  $1.453(6)$  Å from the C(4), C(20), O(3), O(4) plane - the interplanar angle is  $77^\circ$ .

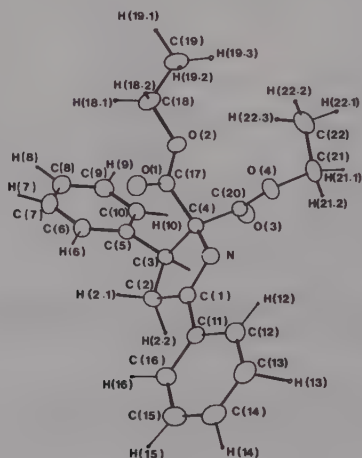


Fig. 1. The  $C_{22}H_{23}NO_4$  molecule.

DIMETHYL 2-((2-((DIETHYLAMINO) CARBONYL)-2-METHYL-3-PHENYL-1-AZIRIDINYL)ETHYLIDENE)-3-OXOBUTANEDIOATE

$C_{22}H_{28}N_2O_6$

M.L.M. PENNINGS, G. OKAY, D.N. REINHOUDT, S. HARKEMA and G.J. VAN HUMMEL, 1982. J. Org. Chem., 47, 4413-4418.

Triclinic,  $P\bar{1}$ ,  $a = 12.034$ ,  $b = 10.878$ ,  $c = 9.181 \text{ \AA}$ ,  $\alpha = 112.31$ ,  $\beta = 103.08$ ,  $\gamma = 88.96^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.038$  for 2133 reflexions.

The analysis established the structure shown in Fig. 1. The relatively short exocyclic carbon-nitrogen bond distance of  $1.365(3) \text{ \AA}$  is nearly the same as the C-N distance of the amide group at C-2 ( $1.346(4) \text{ \AA}$ ), and indicates a partial double bond character through participation of the nitrogen lone-pair electrons. This partial double bond character possibly results in a hindered rotation around the exocyclic C-N bond which explains the broadening in the  $^1H$  NMR spectrum of one of the methoxy signals at  $\delta \sim 3.2$ .

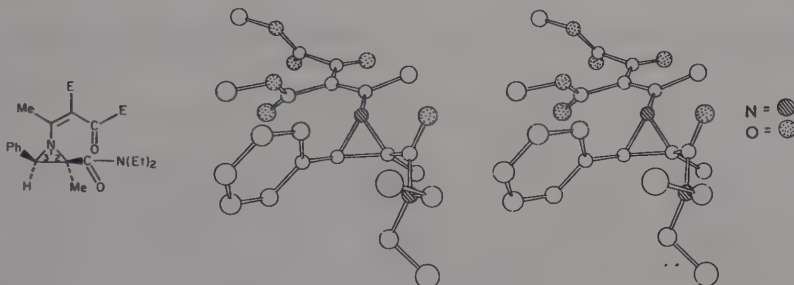


Fig. 1. A stereoview of the aziridine  $C_{22}H_{28}N_2O_6$ .

DIETHYL  $\alpha$ -(1,3-DIPHENYL-2-IMIDAZOLIDINYLIDENE)SUCCINATE

$C_{23}H_{26}N_2O_4$

G. MAAS and R. HOGE, 1982. Cryst. Struct. Comm., 11, 1845-1851.

Monoclinic,  $P2_1/c$ ,  $a = 10.786$ ,  $b = 11.758$ ,  $c = 16.977 \text{ \AA}$ ,  $\beta = 102.38^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.057$  for 2865 reflexions.

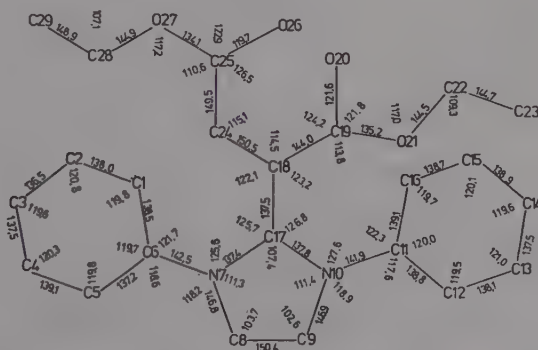


Fig. 1. Bond lengths (pm) and angles for  $C_{23}H_{26}N_2O_4$  ( $\sigma$  0.003 to  $0.006 \text{ \AA}$ ,  $0.2$  to  $0.4^\circ$ ).

The compound (Fig. 1) represents a push-pull substituted alkene. Compounds of this type usually have a highly polarized C=C bond which becomes evident in a bond length substantially longer than in ethylene; here the C=C distance is 1.375(3) Å. The partial reduction in double-bond character allows twisting around the C=C bond (dihedral angles (N,N)C=C(C,C) are -160.9, 24.8, 22.1 and -152.2°).

1-ANILINO-4-ETHOXYCARBONYL-5-METHYL-2,3-DIPHENYL-PYRROLE

$C_{26}H_{24}N_2O_2$  (I)

4-ETHOXYCARBONYL-5-METHYL-2,3-TETRAMETHYLENE-1-(p-NITROANILINO) PYRROLE

$C_{18}H_{21}N_3O_4$  (II)

O. ATTANASI, P. BONIFAZI, E. FORESTI and G. PRADELLA, 1982. J. Org. Chem., **47**, 684-687.

I. Triclinic,  $P\bar{1}$ ,  $a = 12.152$ ,  $b = 10.724$ ,  $c = 8.728$  Å,  $\alpha = 104.90$ ,  $\beta = 99.92$ ,  $\gamma = 88.09^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.088$  for 2438 reflexions.

II. Monoclinic,  $P2_1/a$ ,  $a = 17.08$ ,  $b = 8.34$ ,  $c = 12.40$  Å,  $\beta = 101.33^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.30$  for 1357 reflexions.

The products of the reactions of arylazo-alkenes and  $\beta$ -dicarbonyl compounds, in the presence of copper(II) chloride, have been identified by X-ray analysis. Compound II was decomposed by X-rays and an accurate analysis was not feasible. Compound I is shown in Fig. 1; the ring N-atom is  $sp^2$  hybridised and the pyrrole ring is a nearly regular and perfectly planar pentagon, with only C(2)-N(1)-C(5), at  $111^\circ$ , significantly larger than the  $108^\circ$  value for a regular feature. The normals to the planes of the phenyl groups at C(3) and N(6) are nearly orthogonal with that of the pyrrole ring, while the normal to the plane of the group at C(2) forms an angle of  $126^\circ$  with that normal.

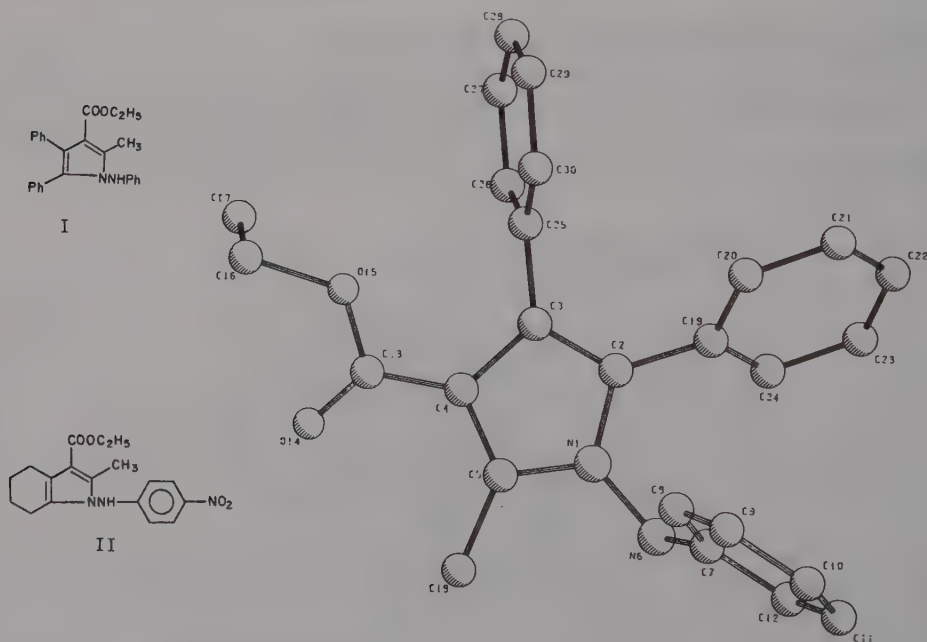
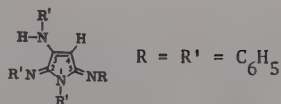


Fig. 1. Formulae of I and II, and a view of a molecule of I.

## N,1-DIPHENYL-2,5-BIS(PHENYLIMINO)-3-PYRROLIN-3-AMINE

 $C_{28}H_{22}N_4$ 

A. ROEDIG, W. RITSCHER, D. SCHEUTZOW and H.J. HECHT, 1982. Chem. Ber., 115, 2652-2663.

Triclinic,  $P\bar{1}$ ,  $a = 11.364$ ,  $b = 10.028$ ,  $c = 11.774$  Å,  $\alpha = 115.57$ ,  $\beta = 77.94$ ,  $\gamma = 113.30^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.058$  for 2750 reflexions.

The structure was confirmed as that shown above. Principal bond lengths are: N(1)-C(2) 1.389(5), N(1)-C(5) 1.428(5), C(2)-C(3) 1.476(6), C(3)-C(4) 1.344(4), C(4)-C(5) 1.450(6), C(2)-N 1.268(5), C(5)-N 1.266(5) Å.

## 3-[3-BENZOYL-4-(DIETHYLAMINO)-5-METHYL-1-PYRAZOLYL]-3-(DIETHYLAMINO)-2-METHYL-1-PHENYL-2-PROPEN-1-ONE

 $C_{29}H_{36}N_4O_2$ 

A. GIEREN and V. LAMM, 1982. Acta Cryst., B38, 844-848.

Monoclinic,  $P2_1$ ,  $a = 8.978$ ,  $b = 16.522$ ,  $c = 19.307$  Å,  $\gamma = 106.07^\circ$ ,  $D_m = 1.135$ ,  $Z = 4$ . Cu radiation,  $R = 0.043$  for 5114 reflexions.

The two molecules (Fig. 1) in the asymmetric unit show some significant differences in conformation and are related in the crystal by an approximate  $n$  glide plane. The  $\pi$ -resonance system of the vinylogous acid amide system of the 2-methyl-1-phenyl-2-propen-1-one residue on N(1) of the pyrazole ring is not planar and shows only a small  $\pi$  interaction with the aromatic pyrazolyl residue. The diethylamino groups have different conformations. The nitrogen of the diethylamino group bound to the pyrazole ring is on the apex of a pyramid. The nitrogen within the vinylogous acid amide system is  $sp^2$ -hybridised and almost coplanar with its bonding neighbours.

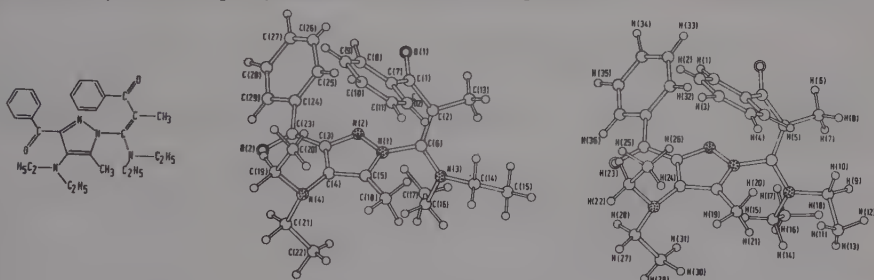


Fig. 1.  $C_{29}H_{36}N_4O_2$ : perspective views of the two molecules in the asymmetric unit.

3,8,12,17-TETRAETHYL-4,5-DIMETHOXY-2,7,13,18-TETRAMETHYL-4,5-DIHYDROBILIN-1,19(21H, 24H)-DIONE n-HEXANE SOLVATE (AETIOBILPURPURIN IV  $\gamma$  n-HEXANE SOLVATE) $C_{33}H_{44}N_4O_4 \cdot 0.5(C_6H_{14})$ 

D.L. CULLEN, N. VAN OPDENBOSCH, E.F. MEYER, JR., K.M. SMITH and F. EIVAZI, 1982. J. Chem. Soc. Perkin II, 307-312.

Triclinic,  $P\bar{1}$ ,  $a = 12.755$ ,  $b = 12.944$ ,  $c = 12.615$  Å,  $\alpha = 112.27$ ,  $\beta = 113.76$ ,  $\gamma = 75.45^\circ$ ,  $D_m = 1.09$ ,  $Z = 2$ . Cu radiation,  $R = 0.105$  for 2298 reflexions.

Rings B, C, and D (Fig. 1) are relatively coplanar with interplanar angles of 5.3 between rings B and C, and  $12.7^\circ$  between C and D, resulting in a quasi-helical



structure for this portion of the molecule. Ring A, linked by  $sp^3$  carbons C(4) and C(5), is considerably out of the plane of the other three rings, making an angle of  $32.3^\circ$  with ring B. The location of the three imino hydrogen atoms suggests that no tautomerism occurs. There is considerable double bond fixation with C(14)-N(23) (1.331) showing more double bond character than C(11)-N(23) (1.386 Å). C(15)-C(16) (1.338) is essentially a double bond while C(14)-C(15) (1.419) has little double bond character. There is an intramolecular hydrogen bond between H(N24) and N(23) of 2.05 Å, but not between H(N22) and N(23) although the N...N distance is similar in both cases. The molecules pack with the relatively planar portions forming layers with an intermolecular hydrogen bond between O(1) and H(N21') of 2.00 Å.

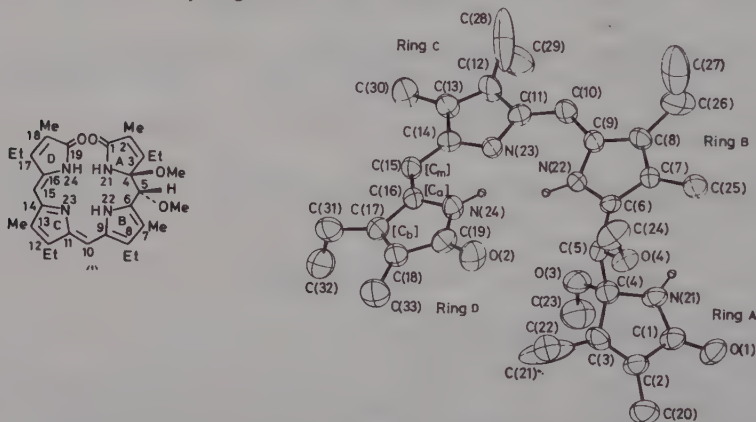


Fig. 1. Molecular skeleton and structure of  $C_{33}H_{44}N_4O_4, 0.5(C_6H_{14})$ .

3,3'-DITHIOBIS(4-BUTYL-1,2-DIPHENYL-3-PYRAZOLINE-5-THIONE)

$C_{38}H_{38}N_4S_4$  (I)

3-ACETYLTHIO-4-BUTYL-3-PYRAZOLINE-5-THIONE

$C_{21}H_{22}N_2OS_2$  (II)

S. SCHEIBYE, A.A. EL-BARBARY, S.-O. LAWESSON, H. FRITZ and G. RIHS, 1982. *Tetrahedron*, **38**, 3753-3760.

I. Monoclinic,  $P2_1/c$ ,  $a = 13.471$ ,  $b = 12.543$ ,  $c = 22.124$  Å,  $\beta = 106.55^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.055$  for 3810 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 10.231$ ,  $b = 9.498$ ,  $c = 21.761$  Å,  $\beta = 100.84^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.072$  for 2887 reflexions.

In I (Fig. 1) the S-S, C=S and C-S distances are 2.073, 1.686 and 1.755 Å respectively. In II, the C=S and C-S distances are 1.681 and 1.793(12) Å.

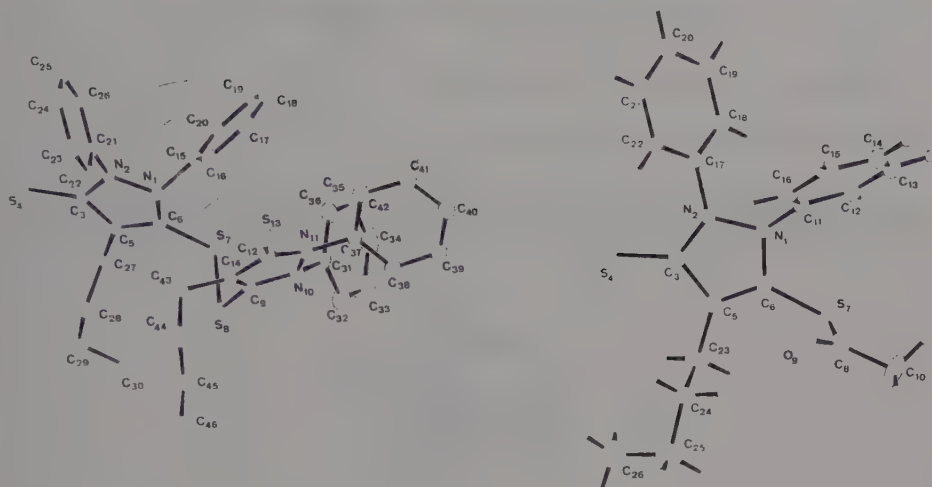


Fig. 1. Views of  $C_{38}H_{38}N_4S_4$  (left) and  $C_{21}H_{22}N_2OS_2$ .

## 2-THIOPYRIDONE

$C_5H_5NS$



U. OHMS, H. GUTH, A. KUTOGLU and C. SCHERINGER, 1982. *Acta Cryst.*, B38, 831-834.

Monoclinic,  $P2_1/c$ ,  $a = 6.107$ ,  $b = 6.325$ ,  $c = 14.382$  Å,  $\beta = 103.0^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.029$  for 884 reflexions; neutron diffraction,  $R = 0.023$  for 949 reflexions.

The crystal structure of 2-thiopyridone has already been determined (1). The molecule (Fig. 1) is essentially planar with the H atom attached to N as deduced previously. Pairs of molecules are linked in the crystal by two N-H...S hydrogen bonds through a crystallographic centre of symmetry with N...S 3.289 Å.

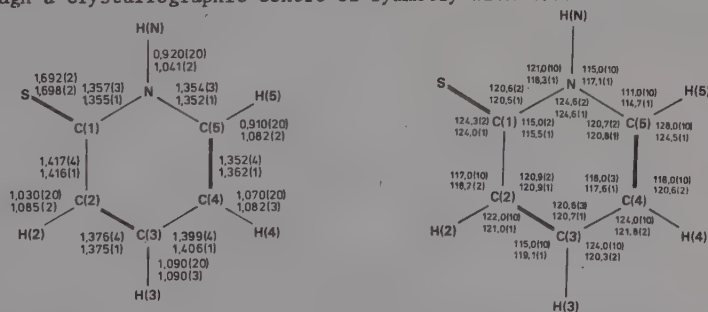


Fig. 1.  $C_5H_5NS$ : bond distances and angles; the upper values refer to the X-ray analysis and the lower values to the neutron diffraction analysis.

1. Structure Reports, 17, 750.

## 1,1,4-TRIMETHYL-1,4-DIHYDRO-1,2,4,5-TETRAZINIUM FLUOROSULPHATE



D.H. HOSKIN, G.P. WOODEN and R.A. OLOFSON, 1982. *J. Org. Chem.*, **47**, 2858-2861.

Orthorhombic,  $P2_12_12_1$ ,  $a = 9.376$ ,  $b = 16.716$ ,  $c = 6.181 \text{ \AA}$ ,  $D_m = 1.5$ ,  $Z = 4$ . Mo radiation,  $R = 0.060$  for 1367 reflexions.

The quaternary nitrogen (N1) is tetrahedral (Fig. 1) which causes the ring to assume a boat shape. There is evidence for substantial delocalization of the N4 electron pair (hybridization between trigonal and tetrahedral; N1 is  $0.51 \text{ \AA}$  above the N2-C3-N5-C6 plane while N4 is only  $0.26 \text{ \AA}$  above the same plane).

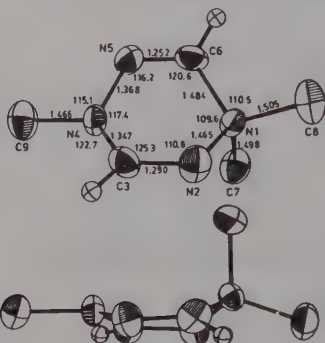
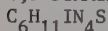


Fig. 1. Bond distances and some angles in the tetrazinium cation.

## 4,5-DIAMINO-1-METHYL-3-(METHYLTHIO)PYRIDAZINIUM IODIDE



B.J. GRAVES and D.J. HODGSON, 1982. *Acta Cryst.*, **B38**, 135-139.

Monoclinic,  $P2_1/n$ ,  $a = 7.549$ ,  $b = 9.605$ ,  $c = 14.568 \text{ \AA}$ ,  $\beta = 89.11^\circ$ ,  $D_m = 1.86$ ,  $Z = 4$ . Mo radiation,  $R = 0.038$  for 2035 reflexions.

The pyridazinium cation (Fig. 1) is planar, with apparently more aromatic character than has been observed in other pyridazine derivatives. Hydrogen bonds link the amino groups and the iodide ions, with  $\text{N}\cdots\text{I}$   $3.712$ ,  $3.710$ ,  $3.668$  and  $3.708 \text{ \AA}$ .

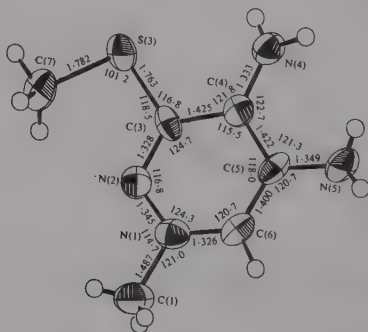


Fig. 1.  $\text{C}_6\text{H}_{11}\text{N}_4\text{S}^+$ : bond distances and angles in the pyridazinium cation.

**trans-2,5-DIMETHYLPIPERAZINE**

K. OKAMOTO, K. SEKIDO, H. ONO, T. NOGUCHI and S. HIROKAWA, 1982. Bull. Chem. Soc. Jpn., 945-946.

Monoclinic,  $P2_1/c$ ,  $a = 8.582$ ,  $b = 5.231$ ,  $c = 8.953$  Å,  $\beta = 118.09^\circ$ ,  $D_m = 1.06$ ,  $Z = 2$ . Mo radiation,  $R = 0.059$  for 1097 reflexions.

The molecule lies about an inversion centre and is in chair conformation (Fig. 1), with mean N-C 1.464, mean C-C 1.521(3) Å. Molecules are linked by weak N-H...N hydrogen bonds (3.305 Å).

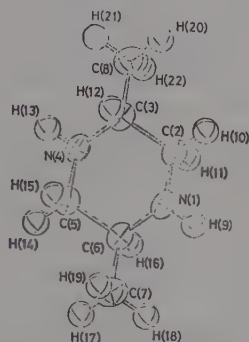
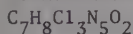


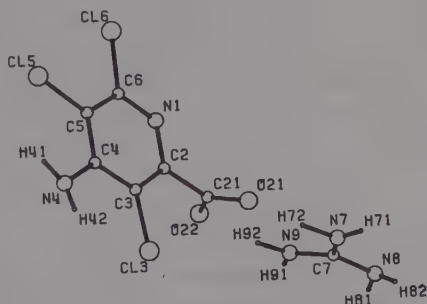
Fig. 1. trans-2,5-dimethylpiperazine.

**GUANIDINIUM 4-AMINO-3,5,6-TRICHLOROPICOLINATE**

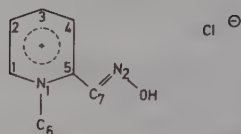
V. PARTHASARATHI, S. WOLFRUM, J.H. NOORDIK, P.T. BEURSKENS, G. SMITH, E.J. O'REILLY and C.H.L. KENNARD, 1982. Cryst. Struct. Comm., 11, 1519-1524.

Monoclinic,  $P2_1/c$ ,  $a = 21.622$ ,  $b = 7.614$ ,  $c = 16.790$  Å,  $\beta = 112.58^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.042$  for 1282 reflexions.

The structure (Fig. 1) contains  $(\text{CH}_6\text{N}_3)^+$  and picolinate anions interlinked by hydrogen bonds. The two independent formula units in the assymmetric unit are related by a pseudo glide plane. In the picolinate ion, the carboxyl group is rotated ( $73.2^\circ$ ,  $73.4^\circ$ ) out of the aromatic ring plane.



2-[(HYDROXYIMINO)METHYL]-1-METHYLPYRIDINIUM CHLORIDE  
 $C_7H_9ClN_2O$



W. VAN HAVERE, A.T.H. LENSTRA, H.J. GEISE, G.R. VAN DEN BERG and H.P. BENSCHOP, 1982. *Acta Cryst.*, **B38**, 2516-2518.

Triclinic,  $P\bar{1}$ ,  $a = 7.110$ ,  $b = 7.165$ ,  $c = 8.861$  Å,  $\alpha = 76.52^\circ$ ,  $\beta = 85.62^\circ$ ,  $\gamma = 65.81^\circ$ ,  $Z = 2$ . Mo radiation,  $R_w = 0.039$  for 2435 reflexions.

In the molecule the oxime moiety has the (E) configuration with C(5)-C(7) 1.460(1), C(7)-N(2) 1.274(1) and N(2)-O 1.372(1) Å. Bond distances in the pyridinium ring, C(1)-C(2) 1.357(1), C(2)-C(3) 1.377(1), C(3)-C(4) 1.365(1) and C(4)-C(5) 1.386(1) Å, suggest that  $\pi$ -localisation is present, as in related compounds. The molecules are stacked in almost perfect planes perpendicular to  $\gamma$ , with no apparent strong interactions between the planes. Within the planes the molecules are linked through hydrogen bonds O-H...Cl<sup>-</sup>, with O...Cl 2.989(1) Å.

5-HYDROXY-6-METHYL-3,4-PYRIDINEDIMETHANOL (PYRIDOXINE)  
 $C_8H_{11}NO_3$

J. LONGO, K.J. FRANKLIN and M.F. RICHARDSON, 1982. *Acta Cryst.*, **B38**, 2721-2724.

Orthorhombic,  $Pn2_1a$ ,  $a = 12.377$ ,  $b = 4.652$ ,  $c = 14.112$  Å,  $D_m = 1.40$ ,  $Z = 4$ . Mo radiation,  $R = 0.048$  for 724 reflexions.

The molecule (Fig. 1) occurs as the non-dipolar form with C(3)-N-C(4)  $119.3(3)^\circ$ , as expected for a non-protonated pyridine ring, and C(2)-O(1) 1.374(4) Å, much longer than the corresponding distances in compounds having a deprotonated phenolic group. Other bond distances and angles are similar to those observed in other structures of vitamin B<sub>6</sub> compounds. An intramolecular hydrogen bond links the phenolic OH group to the adjacent CH<sub>2</sub>OH group, O(1)...O(2) 2.582(5) Å, and intermolecular hydrogen bonds link the molecules in the crystal, O(2)...N' 2.729(5) and O(3)...O(3'') 2.697(6) Å.

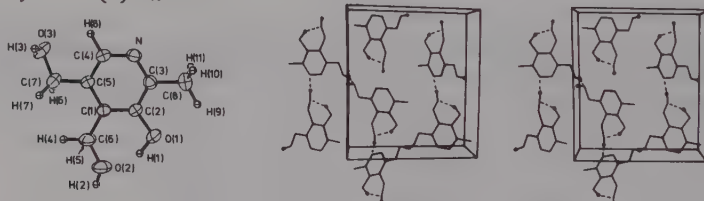


Fig. 1.  $C_8H_{11}NO_3$ : perspective views of the molecule and of the molecular packing; in the latter the  $a$  and  $c$  axes are horizontal and vertical, respectively.

PYRIDOXINE HYDROBROMIDE  
 $C_8H_{12}BrNO_3$  (I)

BIS(PYRIDOXINE-O,O)COPPER TETRACHLOROCOPPER DIHYDRATE  
 $C_{16}H_{26}Cl_4Cu_2N_2O_8$  (II)

V.Kh. SABIROV, Yu.T. STRUCHKOV, A.S. BATANOV and M.A. AZIZOV, 1982. *Koord. Khim.*, **8**, 1623-1628 [*Sov. J. Coord. Chem.*, **8**, 889-894].

I. Triclinic,  $P\bar{1}$ ,  $a = 9.660$ ,  $b = 9.920$ ,  $c = 5.8342$  Å,  $\alpha = 81.57^\circ$ ,  $\beta = 86.10^\circ$ ,  $\gamma = 115.25^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.060$  for 1464 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 10.606$ ,  $b = 8.861$ ,  $c = 7.015$  Å,  $\alpha = 108.86$ ,  $\beta = 99.50$ ,  $\gamma = 105.87^\circ$ ,  $Z = 1$ . Cu radiation,  $R = 0.099$  for 1487 reflexions.

The structure of I comprises pyridoxine cations and bromide anions. The former are protonated at N with N(1)-C(2) and N(1)-C(6) 1.33(1) and 1.34(1) Å respectively (Fig. 1). The pyridine group is planar and C(2'), O(3), C(4') and C(5') lie in this plane. The other substituent atoms deviate from 0.33 to -1.44 Å from this plane. Hydrogen bond distances are: O(3)...O(4') 2.58(1), O(4')...Br 3.188(5), O(5')...Br 3.235(6), N(1)...Br 3.240(5) Å. II contains centrosymmetric complex cations,  $\text{CuCl}_4^-$  anions and waters of hydration. Coordination about Cu is square-planar with Cu-O(3) 1.875(9) and Cu-O(4') 1.980(9) Å. Additional Cu...O (2.545(9)) and Cu...Cl (2.860(3) Å) interactions join the ions to form chains. Pyridoxine chelates Cu by means of the O(3) and O(4') atoms, existing in a bipolar form with a deprotonated O(3) atom and protonated N atom. Coordination of the  $\text{CH}_2\text{OH}$  group results in an increase in the deviation of the O(4') atom from the plane of the pyridine ring (0.3 in I, 0.93 Å in II).

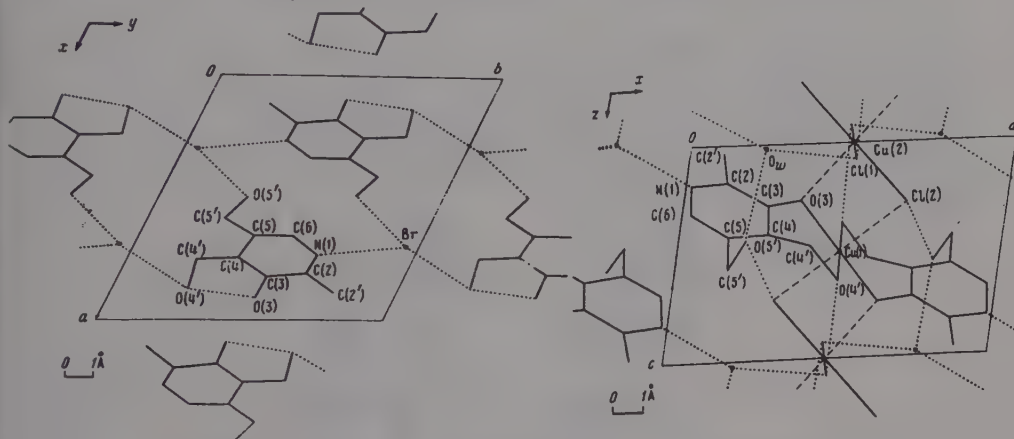


Fig. 1. Projection of the crystal structure of  $\text{C}_8\text{H}_{12}\text{BrNO}_3$  (left) and of  $\text{C}_{16}\text{H}_{26}\text{Cl}_4\text{Cu}_2\text{N}_2\text{O}_8$  (right).

2,2,6,6-TETRAMETHYL-4-OXOPIPERIDINE-1-N-OXIDE (MONOCLINIC FORM)  
 $\text{C}_9\text{H}_{16}\text{NO}_2$

M. LE BARS-COMBE, 1982. Acta Cryst., B38, 2749-2751.

Monoclinic,  $C2/c$ ,  $a = 35.540$ ,  $b = 5.981$ ,  $c = 28.018$  Å,  $\beta = 99.46^\circ$ ,  $Z = 24$ . Cu radiation,  $R_w = 0.06$  for 2123 reflexions (at 173 K).

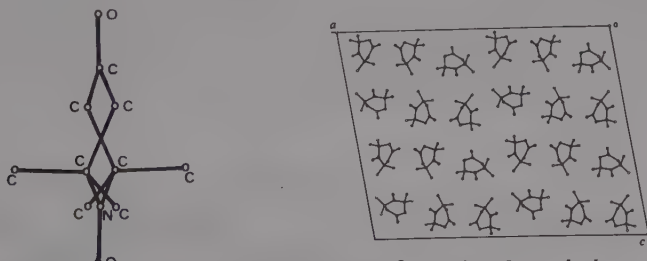


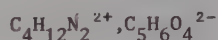
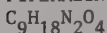
Fig. 1.  $\text{C}_9\text{H}_{16}\text{NO}_2$ : the twist conformation of a molecule and the projection of the crystal structure along  $b$ .



The three independent molecules in the asymmetric unit adopt the twist conformation (Fig. 1). Bond lengths and angles are similar in the three molecules and are in good agreement with those found in the orthorhombic form (1). Intermolecular contacts correspond to van der Waals distances.

1. Structure Reports, 40B, 227; 38B, 262.

PIPERAZINIUM GLUTARATE



M. VANIER and F. BRISSE, 1982. Acta Cryst., B38, 3060-3063.

Triclinic,  $\bar{P}1$ ,  $a = 5.841$ ,  $b = 7.503$ ,  $c = 12.698$  Å,  $\alpha = 97.81^\circ$ ,  $\beta = 99.92^\circ$ ,  $\gamma = 94.89^\circ$ ,  $D_m = 1.35$ ,  $Z = 2$ . Cu radiation,  $R = 0.044$  for 1522 reflexions.

The piperazinium cations (Fig. 1) lie on centres of symmetry and are bonded through hydrogen bonds to the glutarate anions. The torsion angles within the anion correspond to a gauche-trans conformation. The carboxylate groups are tilted by  $15^\circ$  and  $60^\circ$  from their respective methylenic planes. The piperazinium cations are in the chair conformation. A standard geometry is proposed for this cation. Each piperazinium cation is hydrogen-bonded to four different anions and vice versa. The  $\text{N}\cdots\text{O}$  distances range from 2.636(2) to 2.734(3) Å. These hydrogen-bonded structural units form planes parallel to (102).

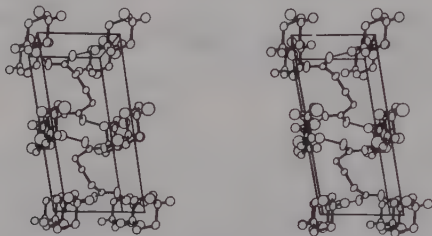
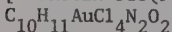


Fig. 1. A stereopair showing the packing of piperazinium glutarate.

[HYDROGEN BIS(PYRIDINE N-OXIDE)] TETRACHLOROGOLD(III)



M.S. HUSSAIN and E.O. SCHLEMPER, 1982. J. Chem. Soc. Dalton, 751-755.

Triclinic,  $\bar{P}1$ ,  $a = 7.276$ ,  $b = 8.631$ ,  $c = 6.799$  Å,  $\alpha = 105.4^\circ$ ,  $\beta = 96.4^\circ$ ,  $\gamma = 67.9^\circ$ ,  $D_m = 2.32$ ,  $Z = 1$ . Mo radiation,  $R = 0.044$  for 1343 reflexions.

The structure comprises discrete  $\text{AuCl}_4^-$  anions and hydrogen bis(pyridine N-oxide) cations with the Au and bridging H atoms located at centres of symmetry (Fig. 1). The hydrogen bond is symmetric with  $\text{O}\cdots\text{O}$  2.41(1) Å. The complex anion is square planar with  $\text{Cl-Au-Cl}$   $89.94(6)^\circ$  and  $\text{Au-Cl}$  2.271(2) Å. There is a short  $\text{Au-O(1)}$  distance (3.105(7) Å). The  $\text{N-O}$  distance in the cation is 1.362(8) Å.

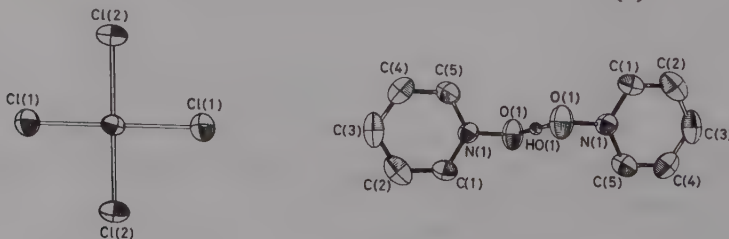
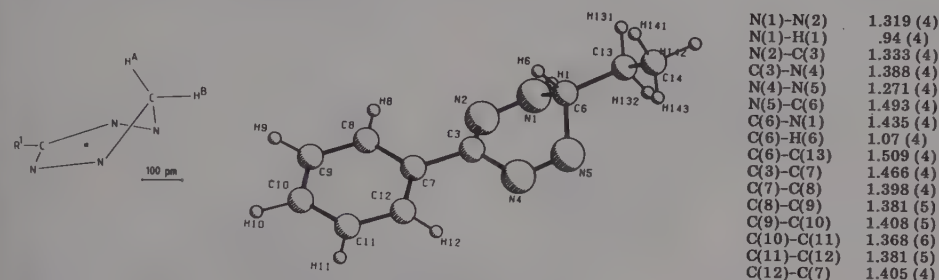


Fig. 1.  $\text{C}_{10}\text{H}_{11}\text{AuCl}_4\text{N}_2\text{O}_2$ : the cation and anion.

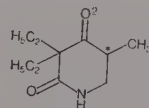
## 6-ETHYL-3-PHENYL-1,6-DIHYDRO-1,2,4,5-TETRAZINE

 $C_{10}H_{12}N_4$ C.H. STAM, A.D. COUNOTTE-POTMAN and H.C. VAN DER PLAS, 1982. *J. Org. Chem.*, **47**, 2856-2858.Orthorhombic,  $Pbca$ ,  $a = 8.349$ ,  $b = 10.252$ ,  $c = 23.041$  Å,  $Z = 8$ . Cu radiation,  $R = 0.053$  for 1454 reflexions (at 233 K).

This analysis revealed that the molecule (Fig. 1) is in a boat conformation with C(6) and C(3) pointing upward with dihedral angles of  $49.3^\circ$  and  $26.7^\circ$ , respectively. N(1) was found to be  $sp^2$  hybridized, and the N(1)-N(2), N(2)-N(3), C(3)-N(4) and N(4)-N(5) bond distances were found to be between single and double bond length, in agreement with the expected electron delocalization.

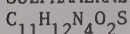
Fig. 1. The  $C_{10}H_{12}N_4$  molecule and bond lengths.

## 3,3-DIETHYL-5-METHYLPYPERIDINE-2,4-DIONE

 $C_{10}H_{17}NO_2$ W.E. OBERHANSLI, 1982. *Helv. Chim. Acta*, **65**, 924-933.Orthorhombic,  $P2_12_12_1$ ,  $a = 13.073$ ,  $12.382$ ,  $12.880$ ,  $12.771$ ,  $b = 11.745$ ,  $12.285$ ,  $11.886$ ,  $11.983$ ,  $c = 6.838$ ,  $6.823$ ,  $6.828$ ,  $6.831$  Å,  $Z = 4$ . Mo radiation,  $R = 0.046$ ,  $0.056$ ,  $0.054$ ,  $0.058$  for 1255, 1217, 1119, 939 reflexions.

Repeated recrystallisations from acetone of a racemic mixture of the title compound successively result in a spontaneous resolution into optical isomers. In this process batches of mixed crystals of increasing optical purity are formed. This phenomenon was investigated on a molecular basis using X-ray diffraction methods. The diffraction patterns of the mixed crystals gave no indication of major disorder. There is a linear relationship between the changes in unit cell parameters  $a$  and  $b$  and the optical rotation of the sample. The molecular shape is such that most of the atomic positions of the two enantiomers superimpose almost perfectly. Occupation factors determined for the asymmetric C-sites agree well with the optical activity of the crystal fraction.

## SULPHAMERAZINE



K.R. ACHARYA, K.N. KUCHELA and G. KARTHA, 1982. *J. Cryst. Spectrosc. Res.*, **12**, 369-376.

Orthorhombic, *Pbca*,  $a = 9.145$ ,  $b = 11.704$ ,  $c = 22.884$  Å,  $D_m = 1.45$ ,  $Z = 8$ . Cu radiation,  $R = 0.078$  for 2082 reflexions.

The sulfamerazine molecule (Fig. 1) is capable of a variety of conformational states by means of rotation about the three bonds C(5)-S, S-N(1), and C(1)-N(1). The three dihedral angles describing these conformations are C(5)-S-N(1)-C(1), S-N(1)-C(1)-N(2), C(6)-C(5)-S-N(1). The values found are 70°, -176°, and 79° respectively. The H-N(1) is eclipsed with N(2) (H(N(1))...N(2) 2.267(10) Å) when projected down the N(1)-C(1) bond. Also it is evident from the structure analysis that rotation about the N(1)-C(1) bond is quite restricted. The pyrimidine and phenyl rings are folded toward each other, making an angle of 104.8°.

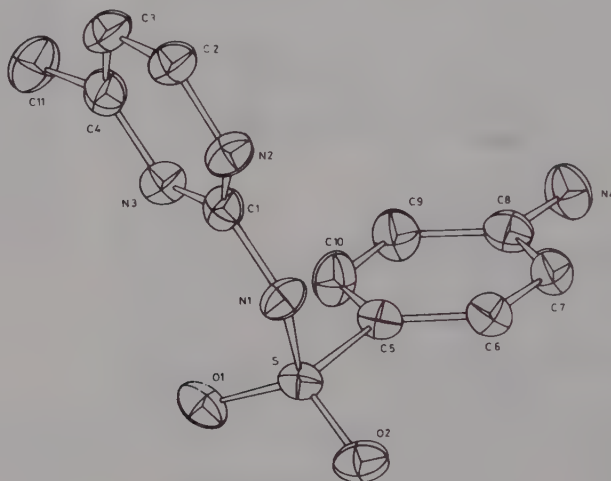
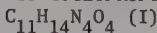
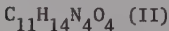


Fig. 1. A view of sulphamerazine.

## cis-CYCLOPROPYLBIS(DIOXOPIPERAZINE)



## trans-CYCLOPROPYLBIS(DIOXOPIPERAZINE)



A. HEMPEL, N. CAMERMAN and A. CAMERMAN, 1982. *J. Am. Chem. Soc.*, **104**, 3456-3458.

I. Orthorhombic, *Pnam*,  $a = 9.731$ ,  $b = 7.080$ ,  $c = 18.208$  Å,  $Z = 4$ . Cu radiation,  $R = 0.049$  for 785 reflexions.

II. Monoclinic, *C2/c*,  $a = 19.172$ ,  $b = 6.650$ ,  $c = 9.854$  Å,  $\beta = 109.43^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.046$  for 757 reflexions.

The structures are shown in Fig. 1, the cis isomer having exact mirror symmetry with the nitrogen atoms eclipsed with respect to the central C-C bond and the trans isomer having exact twofold symmetry. Bond lengths and angles ( $\sigma = 0.002$ - $0.004$  Å,  $0.2$ - $0.3^\circ$ ) are normal. The six-membered rings adopt slightly bowed half-chair conformations.

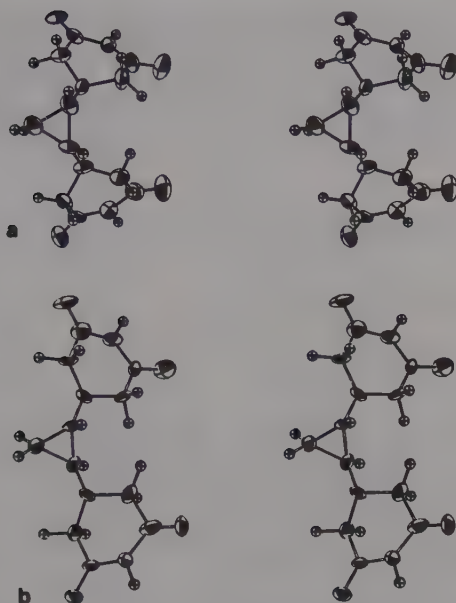


Fig. 1. Stereoviews of cis- (top) and trans-C<sub>11</sub>H<sub>14</sub>N<sub>4</sub>O<sub>4</sub> (bottom).

(±)-4,4'-(1,2-PROPANEDIYL)BIS(4-PIPERAZINE-2,6-DIONE)  
C<sub>11</sub>H<sub>16</sub>N<sub>4</sub>O<sub>4</sub> (I)

S-(+)-4,4'-(1,2-PROPANEDIYL)BIS(4-PIPERAZINE-2,6-DIONE)  
C<sub>11</sub>H<sub>16</sub>N<sub>4</sub>O<sub>4</sub> (II)

A. HEMPEL, N. CAMERMAN and A. CAMERMAN, 1982. J. Am. Chem. Soc., 104, 3453-3456.

I. Triclinic,  $P\bar{1}$ ,  $a = 6.931$ ,  $b = 11.930$ ,  $c = 8.581$  Å,  $\alpha = 101.06$ ,  $\beta = 108.40$ ,  $\gamma = 97.40^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.094$  for 883 reflexions.

II. Monoclinic,  $P2_1$ ,  $a = 10.578$ ,  $b = 9.459$ ,  $c = 6.594$  Å,  $\beta = 95.02^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.041$  for 1126 reflexions.

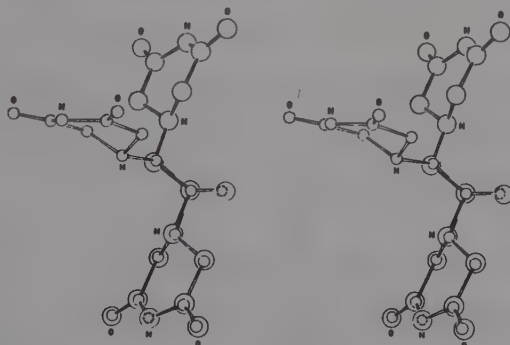


Fig. 1. Stereoviews of racemic (small circles) and S-(+)-C<sub>11</sub>H<sub>16</sub>N<sub>4</sub>O<sub>4</sub> (large circles) superimposed.

The molecular structures of racemic and optically active modifications (I and II respectively) are compared in Fig. 1. The conformations about the central C-C bonds in the two cases are different, I adopting a cis arrangement and II a trans arrangement of the two rings. The conformations of the four piperazinedione rings in I and II are virtually identical, slightly bowed half-chairs in each case. Bond lengths and angles are normal ( $\sigma = 0.01 \text{ \AA}$  and  $1^\circ$  for I,  $0.005 \text{ \AA}$  and  $1^\circ$  for II).

trans-N-(2-PYRIDYLMETHYLENE)ANILINE  
 $C_{12}H_{10}N_2$  (I)

trans-N-(4-PYRIDYLMETHYLENE)ANILINE  
 $C_{12}H_{10}N_2$  (II)

trans-N-BENZYLIDINE-3-PYRIDINAMINE  
 $C_{12}H_{10}N_2$  (III)

M. WIEBCKE and D. MOOTZ, 1982. Acta Cryst., B38, 2008-2013.

I. Monoclinic,  $P2_1/c$ ,  $a = 15.391$ ,  $b = 5.690$ ,  $c = 12.288 \text{ \AA}$ ,  $\beta = 114.17^\circ$ ,  $D_m = 1.24$ ,  $Z = 4$ . Mo radiation,  $R = 0.086$  for 1195 reflexions.

II. Orthorhombic,  $P2_12_12_1$ ,  $a = 5.933$ ,  $b = 7.682$ ,  $c = 21.616 \text{ \AA}$ ,  $D_m = 1.22$ ,  $Z = 4$ . Mo radiation,  $R = 0.073$  for 656 reflexions.

III. Monoclinic,  $P2_1/c$ ,  $a = 10.413$ ,  $b = 8.432$ ,  $c = 21.842 \text{ \AA}$ ,  $\beta = 90.85^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.071$  for 2374 reflexions (at 193 K).

The molecules in all three structures (Fig. 1) are non-planar with torsion angles at the central N and C atoms in the ranges  $8.9$ - $51.3$  and  $-17.9$ - $13.4^\circ$  respectively. The structure of (I) is disordered with two independent molecules lying on centres of symmetry but there is no rotational disorder of the pyridine ring around C(8)-C(9). There are no short intermolecular contacts in any of the structures.

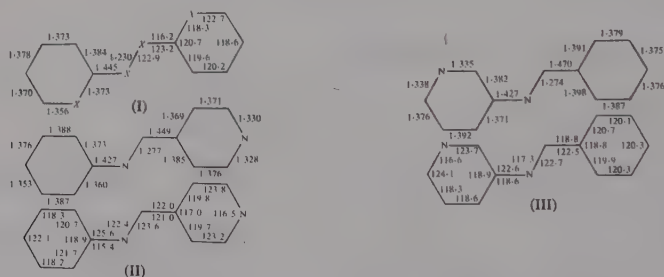


Fig. 1. Bond lengths and angles in the three  $C_{12}H_{10}N_2$  structures. (For I and III average values are given;  $X = 1/2(C + N)$  owing to disorder.)

$\alpha$ -PICOLINE-N-OXIDE HYDROCHLORIDE TRIHYDRATE  
 $C_{12}H_{15}ClN_2O_2 \cdot 3H_2O$

J.C. SPEAKMAN and K.W. MUIR, 1982. Croat. Chem. Acta, 55, 233-248.

Orthorhombic,  $Pccn$ ,  $a = 17.560$ ,  $b = 6.794$ ,  $c = 13.325 \text{ \AA}$ ,  $D_m = 1.296$ ,  $Z = 4$ . Mo radiation,  $R = 0.037$  for 1040 reflexions.

The cation lies across a centre of symmetry, with a very short hydrogen bond ( $O(1) \dots H(O) \dots O(1')$  with  $O \dots O$   $2.414 \text{ \AA}$ ) linking the two halves of the cation (Fig. 1). Bond distances within the cation are: N-O(1)  $1.358$ , N-C(1)  $1.348$ , C(1)-C(2)  $1.387$ , C(2)-C(3)  $1.353$ , C(3)-C(4)  $1.366$ , C(4)-C(5)  $1.360$ , C(5)-N  $1.334$ , C(1)-C(6)

1.476 Å. The anion is shown to be  $\text{Cl}^- \dots \text{H}-\text{OH}$  (or  $\text{HO}-\text{H} \dots \text{Cl}^-$ ) symmetrised by disorder across a twofold axis of the crystal with  $\text{Cl}^- \dots \text{O}$  2.97 Å.

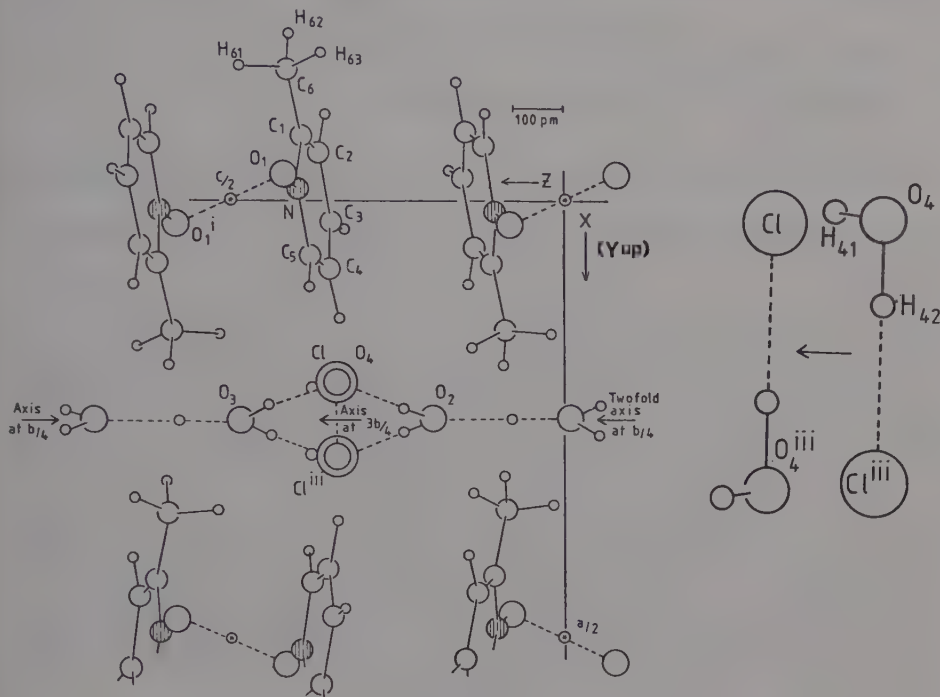


Fig. 1. Crystal structure of  $\text{C}_{12}\text{H}_{15}\text{ClN}_2\text{O}_2 \cdot 3\text{H}_2\text{O}$  viewed down the  $b$  axis (left), and the model for the disordered  $\text{Cl}^- \dots \text{H}-\text{OH}$  unit (arrow indicates the twofold axis) (right).

# CYCLOHEXYL(4-PYRIDYL)METHANOL

$\text{C}_{12}\text{H}_{17}\text{NO}$

N. GALEŠIĆ, I. MATIJAŠIĆ and I. LEBAN, 1982. *Acta Cryst.*, B38, 2275-2277.

Monoclinic,  $\text{P}2_1/\text{c}$ ,  $a = 9.418$ ,  $b = 11.433$ ,  $c = 10.364$  Å,  $\beta = 101.55^\circ$ ,  $D_m = 1.16$ ,  $Z = 4$ . Mo radiation,  $R = 0.077$  for 1077 reflexions.

In the molecule (Fig. 1) the cyclohexyl ring has a slightly distorted chair conformation and the pyridine ring is planar: the dihedral angle between the least-squares planes of these rings is  $79.9(2)^\circ$ . Intermolecular contacts correspond to van der Waals distances and no hydrogen bonds are present in the crystal.

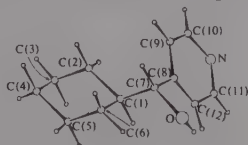
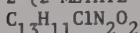


Fig. 1.  $\text{C}_{12}\text{H}_{17}\text{NO}$ : the molecule viewed along  $[010]$ .



## 2-(2-METHYL-3-CHLOROANILINO)NICOTINIC ACID (Four forms)



M. TAKASUKA, H. NAKAI and M. SHIRO, 1982. J. Chem. Soc. Perkin II, 1061-1067.

I. Monoclinic,  $P2_1/c$ ,  $a = 7.625$ ,  $b = 14.201$ ,  $c = 11.672$  Å,  $\beta = 101.65^\circ$ ,  $D_m = 1.40$ ,  $Z = 4$ . Mo radiation,  $R = 0.052$  for 1206 reflexions.

II. Orthorhombic,  $Pca2_1$ ,  $a = 23.597$ ,  $b = 4.042$ ,  $c = 12.127$  Å,  $D_m = 1.50$ ,  $Z = 4$ . Mo radiation,  $R = 0.065$  for 619 reflexions. [Small crystal used for data collection.]

III. Triclinic,  $P\bar{1}$ ,  $a = 13.810$ ,  $b = 3.858$ ,  $c = 10.984$  Å,  $\alpha = 94.98^\circ$ ,  $\beta = 94.42^\circ$ ,  $\gamma = 95.57^\circ$ ,  $D_m = 1.50$ ,  $Z = 2$ . Mo radiation,  $R = 0.070$  for 960 reflexions.

IV. Triclinic,  $P\bar{1}$ ,  $a = 7.670$ ,  $b = 7.254$ ,  $c = 10.882$  Å,  $\alpha = 100.66^\circ$ ,  $\beta = 102.02^\circ$ ,  $\gamma = 86.97^\circ$ ,  $D_m = 1.49$ ,  $Z = 2$ . Mo radiation,  $R = 0.048$  for 1761 reflexions.

The structure of II was determined with less accuracy than the others. In all four forms the carboxy group, N(7), C(8) and the pyrimidine ring (Fig. 1) are almost coplanar. The benzene ring is inclined to this plane at  $70.8$ ,  $40.2$ ,  $21.2$  and  $1.7^\circ$  respectively in forms I-IV. There is increased conjugation throughout the molecule as this angle falls, reflected in the greater double bond character of the N(7)-C(8) bond ( $1.435$ ,  $1.40$ ,  $1.402$  and  $1.402$  Å in I-IV respectively). The electron density map for II indicates that the zwitterionic form is present in the crystal, as is also indicated by the enlargement of the C(2)-N(1)-C(6) angle to  $123$  compared with a mean value of  $118.4^\circ$  for the other forms. There are intramolecular hydrogen bonds between N(7)-H...O(17) in all four forms with N(7)...O(17)  $2.634$ ,  $2.58$ ,  $2.702$  and  $2.667$  Å respectively for I-IV, the value for II again indicating ionisation of the carboxy group. In the crystal structure of I and II there are infinite intermolecularly hydrogen bonded chains extended along the  $b$  and  $c$  axes respectively ( $O(16)...N(1)'$   $2.710$  in I and  $2.75$  Å in II). Dimers, in which two molecules related by an inversion centre are linked by hydrogen bonds, are present in the crystal structures of III and IV, with  $O(16)...O(17)'$   $2.649$  and  $2.694$  Å respectively.

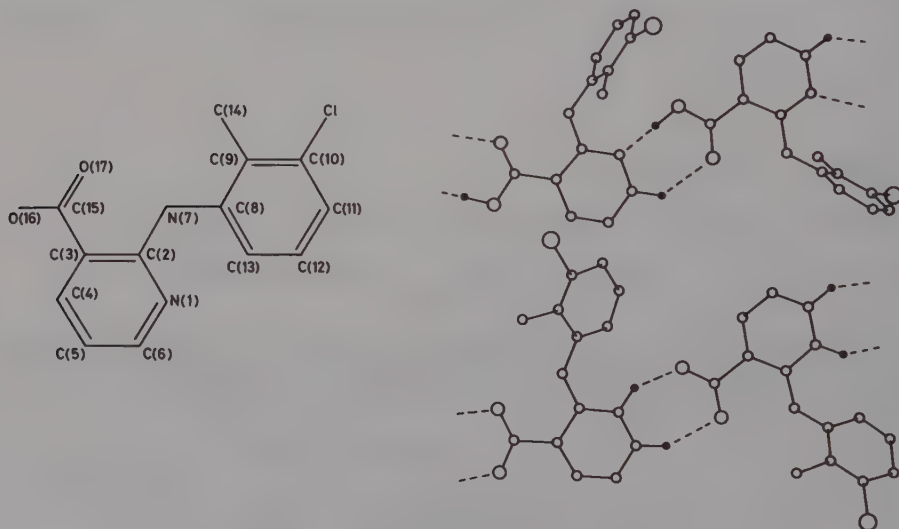
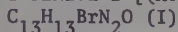
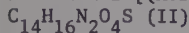


Fig. 1. Molecular skeleton and intermolecular hydrogen bonding in form I (upper right) and form II (lower right) of  $\text{C}_{13}\text{H}_{11}\text{ClN}_2\text{O}_2$ .

## 1-BENZYL-2-[(HYDROXYIMINO)METHYL]PYRIDINIUM BROMIDE



## 1-BENZYL-2-[(HYDROXYIMINO)METHYL]PYRIDINIUM METHYLSULPHONATE



W. VAN HAVERE, A.T.H. LENSTRA, H.J. GEISE, G.R. VAN DEN BERG and H.P. BENSCHOP, 1982. Acta Cryst., B38, 469-472.

I. Orthorhombic, Pcab,  $a = 11.649$ ,  $b = 12.218$ ,  $c = 17.775 \text{ \AA}$ ,  $Z = 8$ . Mo radiation,  $R = 0.041$  for 1634 reflexions.

II. Monoclinic,  $P2_1/b$ ,  $a = 7.044$ ,  $b = 13.113$ ,  $c = 16.50 \text{ \AA}$ ,  $\gamma = 93.10^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.042$  for 1960 reflexions.

In both molecules (Fig. 1) the oxime moiety is in the (E) configuration. The benzyl, pyridinium and oxime parts of both molecules are individually planar. In II, anions and cations are linked by  $\text{O-H}\cdots\text{O}$  ( $2.666(3) \text{ \AA}$ ) hydrogen bonds. In the bromide the corresponding  $\text{Br}\cdots\text{O}(1)$  distance is  $3.128 \text{ \AA}$  which may indicate a similar interaction. The  $\text{C}(7)\text{-N}(2)$  distance is  $1.271(2) \text{ \AA}$  in I and  $1.279(2) \text{ \AA}$  in II.

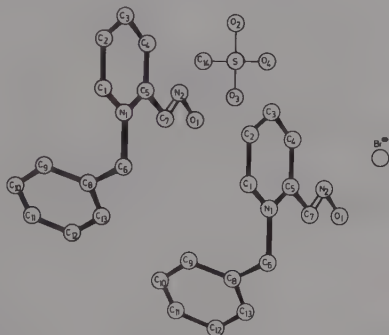


Fig. 1. The structures of the methylsulphonate (II) (left) and bromide (I) (right) pyridinium derivatives.

## ETHYL 2-(METHYLAMINOMETHYLENE)-3-(2-METHYL-6-OXOPIPERIDINO)-3-OXOPROPIONATE



I. CSOREGH, L. PUSZTAY, G. HORVATH and K. SIMON, 1982. Acta Cryst., B38, 3174-3176.

Monoclinic,  $P2_1/c$ ,  $a = 10.322$ ,  $b = 17.218$ ,  $c = 8.698 \text{ \AA}$ ,  $\beta = 112.25^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.058$  for 1949 reflexions.

The piperidine ring (Fig. 1) has a flattened half-chair conformation; the conformation of the side chain is stabilized by an  $\text{N-H}\cdots\text{O}$  hydrogen bond ( $\text{N}\cdots\text{O}$   $2.730(3) \text{ \AA}$ ).

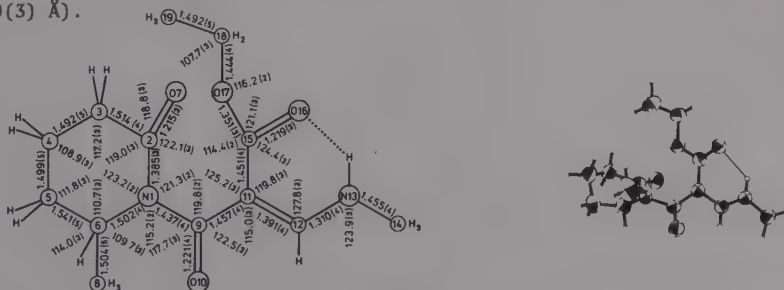
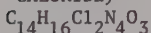


Fig. 1. Molecular dimensions for  $\text{C}_{13}\text{H}_{20}\text{N}_2\text{O}_4$  and a view of the molecule.

1,1'-[OXYBIS(METHYLENE)]BIS{4-[(HYDROXYIMINO)METHYL]PYRIDINIUM} DICHLORIDE (OBIDOXIME CHLORIDE)



W. VAN HAVERE, A.T.H. LENSTRA, H.J. GEISE, G.R. VAN DEN BERG and H.P. BENSCHOP, 1982. Acta Cryst., B38, 1635-1637.

Monoclinic,  $C2/c$ ,  $a = 7.154$ ,  $b = 12.663$ ,  $c = 18.348 \text{ \AA}$ ,  $\beta = 98.07^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.06$  for 756 reflexions.

The two halves of the molecule (Fig. 1), which has the (E,E) configuration at the oxime moieties, are related by a crystallographic twofold axis. The pyridinium and oxime groups are almost planar with the two rings inclined at  $42^\circ$ . Cations and anions are linked by a pair of O-H...Cl hydrogen bonds of length  $2.973(2) \text{ \AA}$ . Some bond lengths are, C(4)-C(7)  $1.422(4)$ , N(2)-O(2)  $1.379(3)$ , C(7)-N(2)  $1.288(3) \text{ \AA}$ .

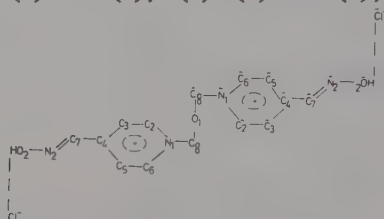
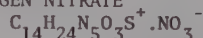
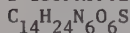


Fig. 1. The molecule of obidoxime chloride.

1-ISOPROPYL-3-{[4-(1-PIPERIDYLAMINO)-3-PYRIDYL]SULPHONYL}UREA HYDROGEN NITRATE



L. DUPONT, O. DIDEBERG, J. DELARGE, G. DIVE and L. THUNUS, 1982. Acta Cryst., B38, 1495-1500.

Monoclinic,  $P2_1/a$ ,  $a = 23.098$ ,  $b = 5.345$ ,  $c = 16.265 \text{ \AA}$ ,  $\beta = 98.51^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.083$  for 2485 reflexions.

The conformation ( $\alpha$ ) of the pyridinesulphonylurea chain (Fig. 1) allows an intramolecular bifurcated hydrogen bond with N(1)...O(1)  $2.767(5)$  and N(1)...O(3)  $2.930(5) \text{ \AA}$  and with angles N-H-O  $131(1)$  and  $110(1)$  and O-H-O  $83(1)^\circ$ . N(2) is protonated with N(2)-C(3)  $1.360(6)$  and N(2)-C(4)  $1.334(6) \text{ \AA}$ . The piperidine ring has the chair form; the angle between its mean plane and that of the pyridine ring is  $79.1(5)^\circ$  and the distance between ring centres is  $5.037(7) \text{ \AA}$ . Molecules are linked by two hydrogen bonds N(2)...O(53)  $2.731(5)$  and N(3)...O(53)  $2.797(5) \text{ \AA}$  involving the nitrate anions. Some bond lengths are C(5)-S  $1.758(4)$ , N(3)-S  $1.620(4)$ , S-O  $1.432(3)$  and  $1.422(3) \text{ \AA}$ .

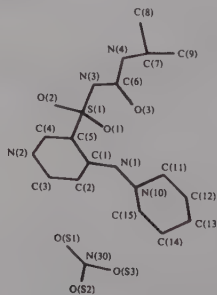
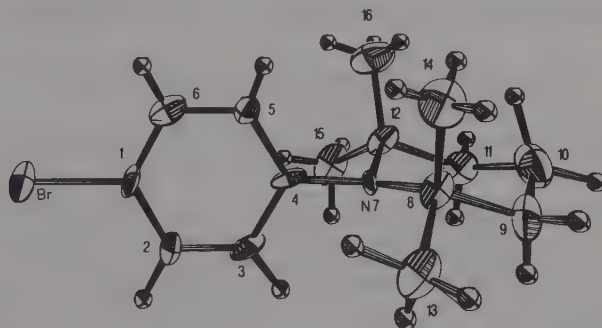


Fig. 1. Atom numbering in  $\text{C}_{14}\text{H}_{24}\text{N}_6\text{O}_6\text{S}$ .

## N-(4-BROMOPHENYL)-2,2,6,6-TETRAMETHYL-PIPERIDINE

 $C_{15}H_{22}BrN$ S. OLEJNIK and R. ALLMANN, 1982. Cryst. Struct. Comm., 11, 1083-1096.Monoclinic,  $P2_1/n$ ,  $a = 12.442$ ,  $b = 16.462$ ,  $c = 7.220$  Å,  $\beta = 90.12^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.0765$  for 1346 reflexions.

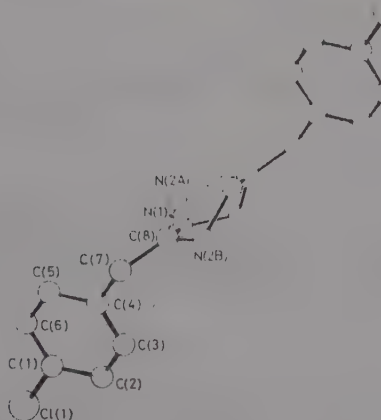
The piperidine ring (Fig. 1) is in the chair-form. The average angle around the nitrogen atom N7 is  $117.1^\circ$ ; N7 lies 0.25 Å away from the plane through the three atoms attached to it. The phenyl ring nearly fits the pseudo-mirror plane of the piperidine ring. The interaction of H5 (at C5) with the axial methyl groups C14 and C16 leads to an increase of the angles N7-C8-C14 and N7-C12-C16 to about  $115.3^\circ$ . The angle N7-C4-C5 is increased too to  $123.9^\circ$ , as compared with N7-C4-C3  $119.0^\circ$ . The H positions at C14 and C15 deviate somewhat from the ideal staggered form. All other atomic distances and bond angles are about normal.

Fig. 1. A view of  $C_{15}H_{22}BrN$ .

## 3,6-BIS(4-CHLOROBENZYL)-1,4-DIHYDRO-s-TETRAZINE

 $C_{16}H_{14}Cl_2N_4$  $(ClC_6H_4CH_2)_2C_2H_2N_4$ 

D. HUNTER, D.G. NEILSON and T.J.R. WEAKLEY, 1982. J. Chem. Soc. Perkin I, 1165-1170.

Monoclinic,  $P2_1/c$ ,  $a = 5.827$ ,  $b = 5.132$ ,  $c = 26.63$  Å,  $\beta = 99.55^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.060$  for 835 reflexions.Fig. 1. Molecular structure of  $C_{16}H_{14}Cl_2N_4$  and alternative conformations for the dihydrotetrazine ring.

The X-ray structure confirms that this compound is the 1,4-dihydro isomer (Fig. 1), as the C(8)-N(1) bond is double (1.27 Å) while the other bonds in the heterocyclic ring are single. The molecule is partly disordered in the crystal, with the chlorophenyl groups appearing fixed while N(2) lies at two sites, N(2A) and N(2B). C(7), C(8) and N(1) have high anisotropic thermal parameters but alternate sites can not be distinguished. The disorder involves two equally probable acentric boat conformations for the dihydrotetrazine ring, and these two conformations are crystallographic centrosymmetrically related sets. N(2B), C(8), N(1) and N(2A') are almost coplanar, and the intermolecular distances N(1)...N(2A) (2.97) and N(1)...N(2B) (2.99 Å) are consistent with N-H...N hydrogen bonds.

2,6-DIMETHYL-3,5-DICARBOMETHOXY-4-PHENYL-1,4-DIHYDROPYRIDINE  
 $C_{17}H_{19}NO_4$  (I)

2,6-DIMETHYL-3,5-DICARBOMETHOXY-4-(3-NITROPHENYL)-1,4-DIHYDROPYRIDINE  
 $C_{17}H_{18}N_2O_6$  (II)

2,6-DIMETHYL-3,5-DICARBOMETHOXY-4-(4-NITROPHENYL)-1,4-DIHYDROPYRIDINE  
 $C_{17}H_{18}N_2O_6$  (III)

2,6-DIMETHYL-3,5-DICARBOMETHOXY-4-(2,4-DINITROPHENYL)-1,4-DIHYDROPYRIDINE  
 $C_{17}H_{17}N_3O_8$  (IV)

2,6-DIMETHYL-3,5-DICARBOMETHOXY-4-(4-METHYLPHENYL)-1,4-DIHYDROPYRIDINE  
 $C_{18}H_{21}NO_4$  (V)

2,6-DIMETHYL-3,5-DICARBOMETHOXY-4-(3-METHYLPHENYL)-1,4-DIHYDROPYRIDINE  
 $C_{18}H_{21}NO_4$  (VI)

R. FOSSHEIM, K. SVARTENG, A. MOSTAD, C. RØMMING, E. SHEFTER and D.J. TRIGGLE, 1982. J. Med. Chem., **25**, 126-131.

I. Triclinic,  $P\bar{1}$ ,  $a = 7.376$ ,  $b = 9.596$ ,  $c = 11.663$  Å,  $\alpha = 110.54^\circ$ ,  $\beta = 95.08^\circ$ ,  $\gamma = 99.78^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.065$  for 1507 reflexions (at  $-150^\circ\text{C}$ ).

II. Monoclinic,  $P2_1/n$ ,  $a = 14.833$ ,  $b = 7.390$ ,  $c = 15.685$  Å,  $\beta = 109.92^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.107$  for 2246 reflexions (at  $-150^\circ\text{C}$ ).

III. Monoclinic,  $P2_1/c$ ,  $a = 10.495$ ,  $b = 16.000$ ,  $c = 10.702$  Å,  $\beta = 115.80^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.032$  for 4308 reflexions (at  $-150^\circ\text{C}$ ).

IV. Monoclinic,  $P2_1/c$ ,  $a = 11.800$ ,  $b = 10.136$ ,  $c = 15.050$  Å,  $\beta = 109.27^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.046$  for 4089 reflexions (at  $-150^\circ\text{C}$ ).

V. Monoclinic,  $P2_1/c$ ,  $a = 10.244$ ,  $b = 16.016$ ,  $c = 10.709$  Å,  $\beta = 114.93^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.075$  for 3793 reflexions (at  $-150^\circ\text{C}$ ).

VI. Monoclinic,  $P2_1/c$ ,  $a = 15.106$ ,  $b = 7.347$ ,  $c = 17.392$  Å,  $\beta = 123.47^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.052$  for 5979 reflexions (at  $-150^\circ\text{C}$ ).

The dihydropyridine ring in each compound exists in a boat-type conformation (Fig. 1). The degree of ring puckering varies among the compounds, and the distortions are influenced to a great extent by the position of the substituent in the 4-phenyl ring and the conformation about the inter-ring bond. The distortion at the apical N of the dihydropyridine ring was found to be linearly related to that at the apical tetrahedral carbon.

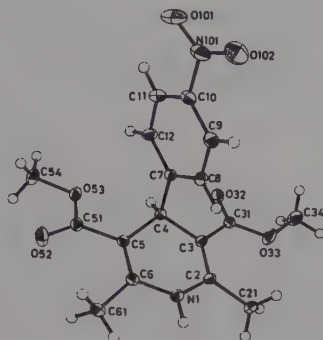


Fig. 1. Molecular structure of  $C_{17}H_{18}N_2O_6$  (III).

## 1,4-DIACETYL-1,4-DIHYDRO-2,3-DIPHENYLPIPERAZINE

$$\text{C}_{20}\text{H}_{18}\text{N}_2\text{O}_2$$

J. ARMAND, C. BOIS, M. PHILOCHE-LEVISALLES, M.-J. POUET and M.-P. SIMONNIN, 1982. *Canad. J. Chem.*, 60, 349-354.

Triclinic,  $P\bar{1}$ ,  $a = 5.684$ ,  $b = 9.299$ ,  $c = 16.516$  Å,  $\alpha = 89.62$ ,  $\beta = 90.16$ ,  $\gamma = 107.29^\circ$ ,  $D_m = 1.26$ ,  $Z = 2$ . Mo radiation,  $R = 0.043$  for 2114 reflexions.

The analysis shows that the 1,4-dihydropiperazine skeleton has a boat conformation (Fig. 1); the C2-C3 and C5-C6 double bonds are localized.

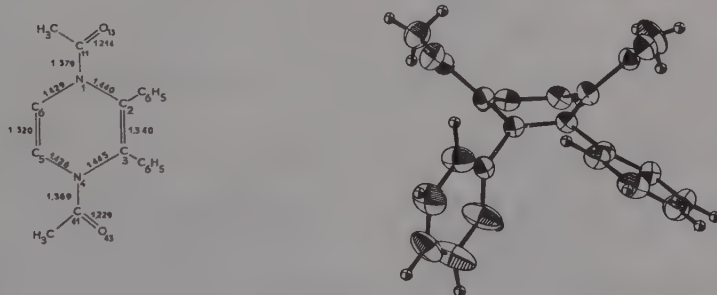


Fig. 1. Bond lengths in  $C_{20}H_{18}N_2O_2$  and a view showing the boat conformation of the 1,4-dihydropiperazine ring.

4-[4-(4-BROMOPHENYL)-4-HYDROXYPIPERIDINO]-4'-FLUOROBUTYROPHENONE (BROMPERIDOL)

$$\text{C}_{21}\text{H}_{23}\text{BrFNO}_2$$

B. TINANT, G. GERMAIN, J.P. DECLERCQ, M. VAN MEERSSCHE, M. AZIBI, M. DRAGUET-BRUGHMANS and R. BOUCHE, 1982. *Cryst. Struct. Comm.*, 11, 633-638.

Monoclinic,  $P2_1/c$ ,  $a = 7.741$ ,  $b = 9.020$ ,  $c = 28.973$  Å,  $\beta = 106.23^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.057$  for 1366 reflexions.

The piperidine ring is in chair conformation and its mean plane is perpendicular to the mean plane of the bromophenyl ring (Fig. 1). The hydroxyl occupies a transoid position with respect to the nitrogen lone-pair. Molecules are linked by O-H...N hydrogen bonds (O...N 2.867 Å).



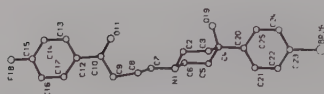


Fig. 1. The bromperidol molecule.

4'-FLUORO-4-(4-HYDROXY-4-PHENYLPYPERIDINO)BUTYROPHENONE  
 $C_{21}H_{24}FNO_2$

B. TINANT, G. GERMAIN, J.P. DECLERCQ, M. VAN MEERSCHE, M. AZIBI, M. DRAGUET-BRUGHMANS and R. BOUCHE, 1982. Cryst. Struct. Comm., 11, 639-645.

Monoclinic,  $P2_1/c$ ,  $a = 16.923$ ,  $b = 9.175$ ,  $c = 13.794$  Å,  $\beta = 121.53^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.045$  for 1598 reflexions.

The molecule conformation is shown in Fig. 1. The piperidine ring is in chair form, with its mean plane perpendicular to the mean plane of the adjacent phenyl ring. Molecules are linked by O-H...N hydrogen bonds ( $O...N$  2.94 Å).

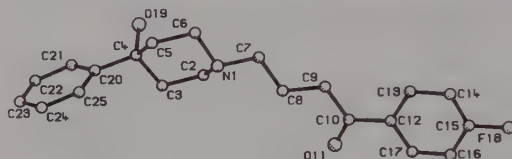


Fig. 1. The  $C_{21}H_{24}FNO_2$  molecule.

1-(1-PHENYL-t-4-tert-BUTYL-r-CYCLOHEXYL)PIPERIDINE HYDROCHLORIDE  
 $C_{21}H_{34}ClN$

P. BRIARD, R. ROQUES, J.M. KAMENKA, P. GENESTE, J.P. DECLERCQ and G. GERMAIN, 1982. Cryst. Struct. Comm., 11, 231-234.

Monoclinic,  $P2_1/c$ ,  $a = 11.418$ ,  $b = 13.528$ ,  $c = 13.309$  Å,  $\beta = 99.84^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.053$  for 2195 reflexions.

The presence of the t-butyl group in the molecule (Fig. 1) leads to only a slight modification of the structure of the parent molecule (1).

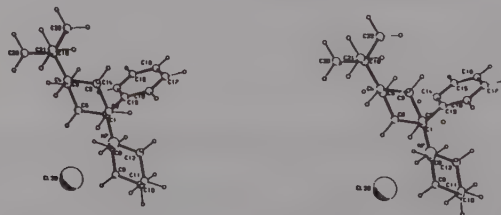


Fig. 1. A stereoview of  $C_{21}H_{34}ClN$ .

1. Structure Reports, 35B, 176.

[2-(4-(DIPHENYLMETHYL)-1-PIPERIDINYL)-2-OXOETHOXY]ACETIC ACID  
 $C_{22}H_{25}NO_4$  (I)

DICALCIUM TETRAKIS{[2-(4-(DIPHENYLMETHYL)-1-PIPERIDINYL)-2-OXOETHOXY]ACETATE}  
 HYDRATE

$C_{88}H_{96}Ca_2N_4O_{16} \cdot 10.8(H_2O)$  (II)  $[Ca(C_{22}H_{24}NO_4)_2]_2 \cdot 10.8(H_2O)$

P. VAN ROEY, G.D. SMITH, W.L. DUAX, M.J. UMEN and B.E. MARYANOFF, 1982. J. Am. Chem. Soc., 104, 5661-5666.

I. Monoclinic,  $P2_1/c$ ,  $a = 20.244$ ,  $b = 5.822$ ,  $c = 16.363$  Å,  $\beta = 95.85^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.058$  for 2650 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 17.460$ ,  $b = 18.089$ ,  $c = 15.834$  Å,  $\alpha = 97.24$ ,  $\beta = 88.24$ ,  $\gamma = 113.74^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.071$  for 11496 reflexions.

The structure of the free acid I is shown in Fig. 1 and that of each of the two independent centrosymmetric binuclear Ca complexes (which each contain two  $H_2O$  molecules) in Fig. 2. The Ca ions are 8-coordinate with square antiprismatic geometry (Ca-O 2.381-2.581(3-5) Å). The two complexes of II are stereoisomers but have very similar overall geometry. In contrast to the free acid, the ionophores in II have nearly planar diglycolamic moieties. The dimers are held together through bridging carboxylate groups of two of the ionophores.

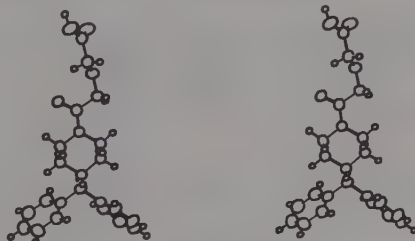


Fig. 1. Stereoview of  $C_{22}H_{25}NO_4$ .

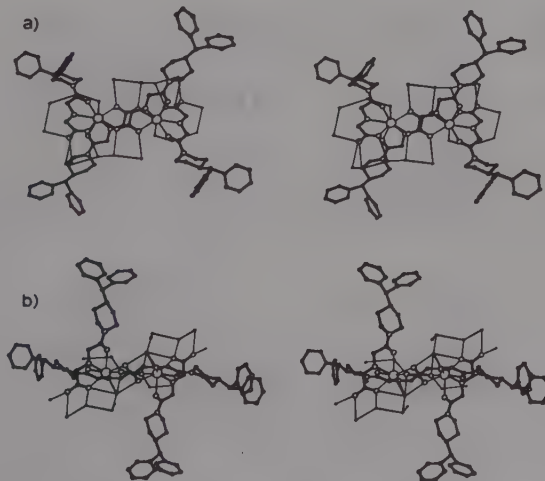


Fig. 2. Stereoviews of the two independent complexes  $[Ca(C_{22}H_{24}NO_4)_2]_2 \cdot 10.8(H_2O)$ .

1-(4-FLUOROPHENYL)-4-[4-HYDROXY-4-(4-METHYLPHENYL)-1-PIPERIDINYL]-1-BUTANONE  
(MOPERONE)

$C_{22}H_{26}FNO_2$  (I)

1-(4-FLUOROPHENYL)-4-[4-HYDROXY-4-(4-METHYLPHENYL)-1-PIPERIDINYL]-1-BUTANONE HYDRO-  
BROMIDE

$C_{22}H_{27}BrFNO_2$  (II)

N.M. BLATON, O.M. PEETERS and C.J. DE RANTER, 1982. Cryst. Struct. Comm., **11**, 1283-1292.

I. Monoclinic,  $P2_1/a$ ,  $a = 10.859$ ,  $b = 21.44$ ,  $c = 16.984$  Å,  $\beta = 96.99^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.057$  for 1625 reflexions. [See also the following Report.]

II. Monoclinic,  $P2_1/c$ ,  $a = 17.514$ ,  $b = 8.905$ ,  $c = 14.580$  Å,  $\beta = 106.73^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.058$  for 1644 reflexions.

In both structures the piperidine ring is in chair conformation with an axial hydroxyl and equatorial phenyl moiety (Fig. 1). The two independent molecules of the free base have the butyrophenone side chain fully extended; in the salt there is a rotation of about  $90^\circ$  around the C17-C18 bond. Packing corresponds to van der Waals interactions in the free base; in the salt there is O-H...O hydrogen bonding ( $O...O$  2.851(6) Å).

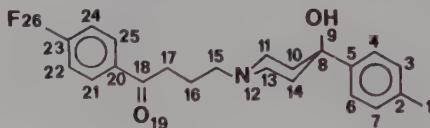


Fig. 1. The moperone free base.

MOPERONE

$C_{22}H_{26}FNO_2$

B. TINANT, G. GERMAIN, J.P. DECLERCQ, M. VAN MEERSSCHE, M. AZIBI, M. DRAGUET-BRUGHMANS and R. BOUCHÉ, 1982. Bull. Soc. Chim. Belg., **91**, 283-287.

Monoclinic,  $P2_1/c$ ,  $a = 16.907$ ,  $b = 21.363$ ,  $c = 10.825$  Å,  $\beta = 96.87^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.105$  for 1239 reflexions. [See also the preceding Report.]

A view of the two molecules in the asymmetric unit is given in Fig. 1. The major difference between the two molecules is that in one molecule, there are columns of  $O(19)-H...N1$  intermolecular hydrogen bonds (2.85 Å), whereas in the other there are  $O(19)-H...C(22)$  (3.37 Å) to form dimers.

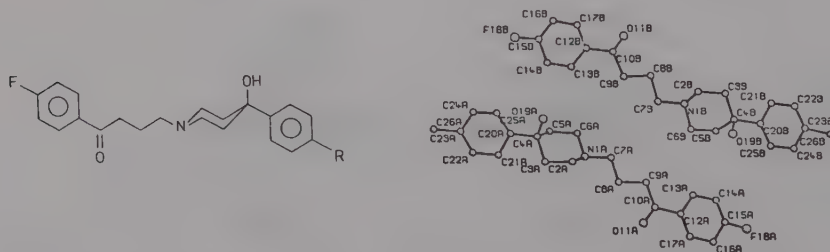
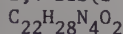


Fig. 1. The moperone molecules ( $R = CH_3$ ).

## 1,4-BIS(2-SALICYLIDENEAMINOETHYL)PIPERAZINE



B. CHIARI, O. PIOVESANA, T. TARANTELLI and P.F. ZANAZZI, 1982. Acta Cryst., B38, 331-333.

Monoclinic,  $P2_1/a$ ,  $a = 13.328$ ,  $b = 11.654$ ,  $c = 6.384 \text{ \AA}$ ,  $\beta = 94.02^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.048$  for 956 reflexions.

The molecules (Fig. 1) lie on inversion centres. The piperazine ring has the chair conformation whilst the benzene ring is planar. Bond distances and angles are close to expected values with mean  $\text{C}(\text{sp}^3)\text{-C}(\text{sp}^3)$  1.507, mean  $\text{C-N}$  (single bond) 1.460,  $\text{C=N}$  1.265 and  $\text{C-O}$  1.350  $\text{\AA}$ . Intermolecular  $\text{O-H}\cdots\text{N}$  hydrogen bonds with  $\text{O}\cdots\text{N}$  2.589  $\text{\AA}$  are present. Other intermolecular contacts correspond to van der Waals interactions.

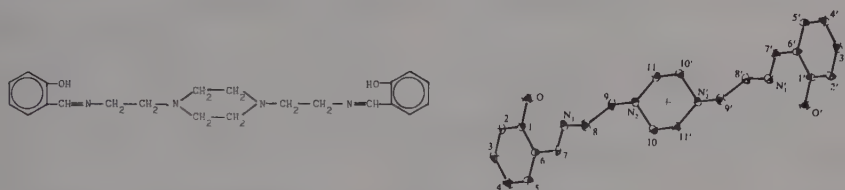
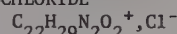
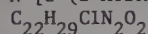


Fig. 1. A perspective view of the  $\text{C}_{22}\text{H}_{28}\text{N}_4\text{O}_2$  molecule.

## N-[1-(2-HYDROXY-2-PHENYLETHYL)-4-PIPERIDYL]-N-PHENYLPROPANAMIDE HYDROCHLORIDE



A. MICHEL, B. LEBRUN, G. EVRARD and F. DURANT, 1982. Acta Cryst., B38, 2961-2963.

Monoclinic,  $P2_1/c$ ,  $a = 12.104$ ,  $b = 11.560$ ,  $c = 15.013 \text{ \AA}$ ,  $\beta = 101.14^\circ$ ,  $D_m = 1.25$ ,  $Z = 4$ . Cu radiation,  $R = 0.06$  for 2538 reflexions.

In the molecule (Fig. 1) the N side chain of the piperidyl group is in a pseudo-equatorial position. This moiety adopts an extended conformation. Packing is governed by van der Waals interactions; the  $\text{Cl}^-$  ion does not contribute to the formation of intermolecular hydrogen bonds

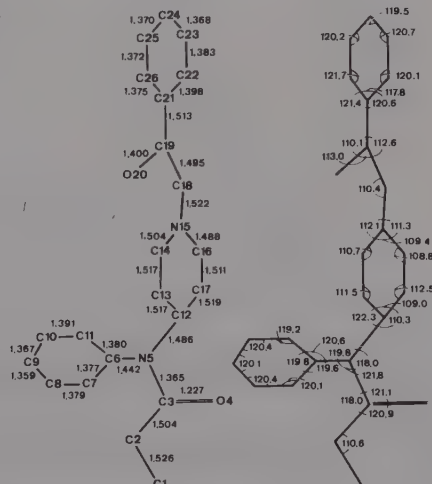
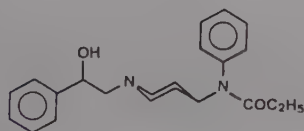


Fig. 1.  $\text{C}_{22}\text{H}_{29}\text{ClN}_2\text{O}_2$ : structural formula and bond lengths and angles in the molecule.

N-BENZYL-3-t-BUTYL-5-FORMYL-2-HYDROXY-2-PIVALOYL-1,2-DIHYDROPYRIDINE  
 $C_{22}H_{29}NO_3$



A. NISHINAGA, T. SHIMIZU, Y. TOYODA, T. MATSUURA and K. HIROTSU, 1982. *J. Org. Chem.*, **47**, 2278-2285.

Monoclinic,  $C2/c$ ,  $a = 22.858$ ,  $b = 8.579$ ,  $c = 21.812$  Å,  $\beta = 107.12^\circ$ ,  $D_m = 1.14$ ,  $Z = 8$ .  
 Mo radiation,  $R = 0.054$  for 1757 reflexions.

The analysis establishes the structure as shown above ( $R = CH_2Bz$ ).

trans-4'-HYDROXY-N-METHYL-4-STILBAZOLIUM (+)-CAMPHOR-10-SULPHONATE

$C_{24}H_{29}NO_5S$

$C_{14}H_{14}NO^+$ ,  $C_{10}H_{15}O_4S^-$

R.F. ZIOLO, W.H.H. GÜNTHER, G.R. MEREDITH, D.J. WILLIAMS and J.M. TROUP, 1982.  
*Acta Cryst.*, **B38**, 341-343.

Monoclinic,  $P2_1$ ,  $a = 8.475$ ,  $b = 28.192$ ,  $c = 9.605$  Å,  $\beta = 98.28^\circ$ ,  $D_m = 1.29$ ,  $Z = 4$ .  
 Cu radiation,  $R = 0.044$  for 3096 reflexions.

In the crystal structure two crystallographically independent hydrogen-bonded cation-anion pairs (Fig. 1) with  $O(1)...O(7)$  2.595 Å and  $O(2)...O(3)$  2.616 Å, interleave to form dimers in layers normal to (010). In the dimer the cations are related by a pseudo inversion centre and have a non-coplanar antiparallel arrangement.

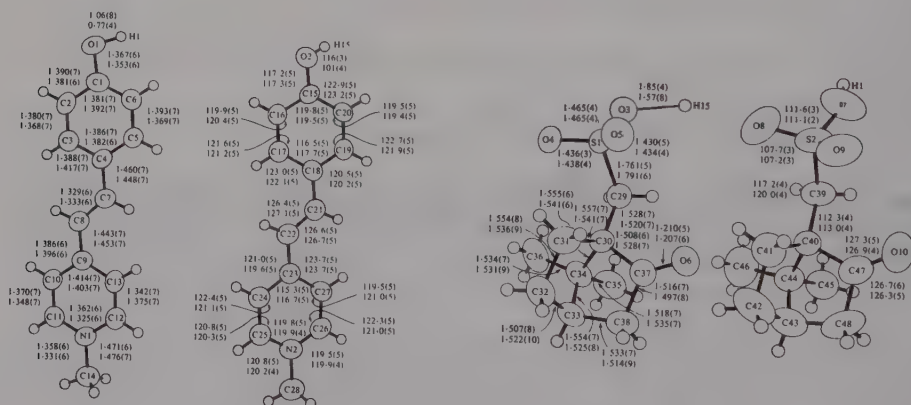


Fig. 1. Bond distances and angles in the two crystallographically independent 4'-hydroxy-N-methyl-4-stilbazolium cations and the (+)-camphor-10-sulphonate anions; in each case upper numbers refer to the labelled ion, while lower numbers refer to the corresponding value in the other ion.

2,6-DIMETHYL-4-(3-NITROPHENYL)-3,5-BIS(NEOPENTYLOXYCARBONYL)-1,4-DIHYDROPYRIDINE  
 $C_{25}H_{34}N_2O_6$  (I)

2,6-DIMETHYL-4-(3-NITROPHENYL)-3-(NEOPENTYLOXYCARBONYL)-5-(TRIMETHYLSILYLMETHOXY-CARBONYL)-1,4-DIHYDROPYRIDINE  
 $C_{24}H_{34}N_2O_6Si$  (II)

R. TACKE, A. BENTLAGE, W.S. SHELDRIK, L. ERNST, R. TOWART and K. STOEPEL, 1982.  
*Z. Naturforsch.*, **37b**, 443-450.

I. Monoclinic,  $P2_1/c$ ,  $a = 12.268$ ,  $b = 10.689$ ,  $c = 20.153$  Å,  $\beta = 106.74^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.069$  for 2741 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 12.457$ ,  $b = 10.835$ ,  $c = 20.436$  Å,  $\beta = 106.95^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.092$  for 4006 reflexions.

Molecules I and II are isostructural (Fig. 1) with normal values for bond lengths and angles. The heterocycle adopts a tub conformation and the phenyl ring is nearly planar to it (dihedral angle  $88.0(4)$ ,  $84.7(4)^\circ$  for I and II respectively). Both carbonyl groups are arranged antiperiplanar to the C(3)-C(4) and C(5)-C(4) bonds.

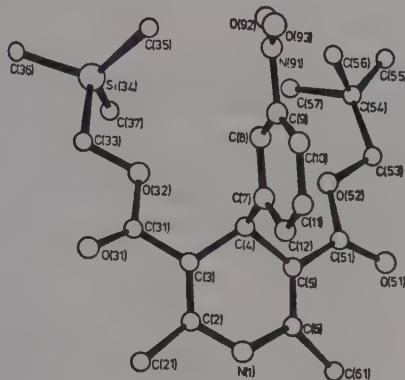


Fig. 1. Perspective view of  $C_{24}H_{34}N_2O_6Si$  (II) (the structure I is similar with C replacing the Si atom).

(-)-2-[4-[2-HYDROXY-2-(3',4'-DIMETHYLPHENYL)ETHYL]-1-PIPERAZINYL]-2,4,6-CYCLOHEPTATRIEN-1-ONE p-BROMOBENZOATE

$C_{28}H_{29}BrN_2O_5$

F.R. AHMED and J. BAGLI, 1982. *Canad. J. Chem.*, **60**, 2687-2691.

Monoclinic,  $P2_1$ ,  $a = 12.553$ ,  $b = 6.359$ ,  $c = 17.575$  Å,  $\beta = 108.32^\circ$ ,  $D_m = 1.384$ ,  $Z = 2$ . Cu radiation,  $R = 0.036$  for 2155 reflexions.

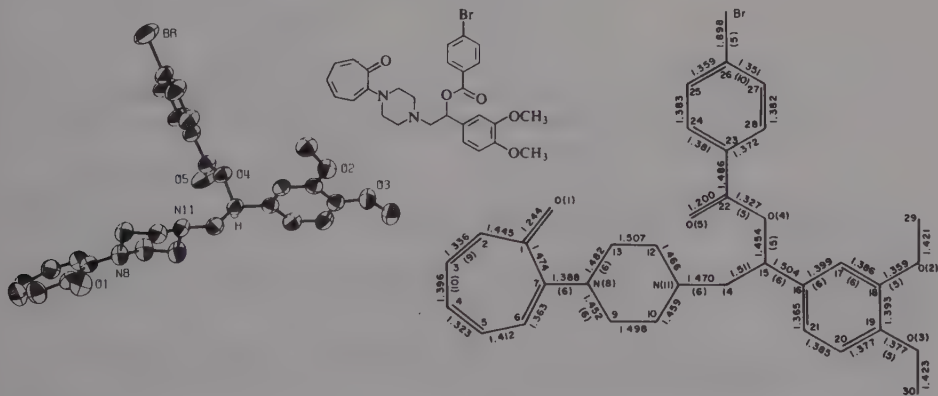


Fig. 1. A view of the  $C_{28}H_{29}BrN_2O_5$  molecule and bond lengths. Esd's are 0.007-0.008 Å except for those given in parentheses.



The piperazine ring (Fig. 1) has a distorted chair form that is flattened at N8. The cycloheptatriene ring is nearly planar.

# PIPERAZINIUM BIS(n-DODECANOATE)

$C_{28}H_{58}N_2O_4$

$2C_{12}H_{23}O_2^-, C_4H_{12}N_2^{2+}$

F. BRISSE and J.-P. SANGIN, 1982. Acta Cryst., B38, 215-221.

Triclinic,  $P\bar{1}$ ,  $a = 5.680$ ,  $b = 7.456$ ,  $c = 18.576$  Å,  $\alpha = 84.22^\circ$ ,  $\beta = 82.67^\circ$ ,  $\gamma = 71.67^\circ$ ,  $D_m = 1.048$ ,  $Z = 1$ . Mo radiation,  $R = 0.044$  for 938 reflexions.

The crystal structure (Fig. 1) consists of a piperazinium cation, in the chair conformation, situated on a crystallographic centre of symmetry, and two centrosymmetrically related n-dodecanoate anions. The anion does not have the fully-extended (all-trans) conformation, since a torsion angle of  $78^\circ$  is observed around the C(3)-C(4) bond. However, the C(1)-C(4) and C(3)-C(12) moieties remain planar. The dihedral angle between the carboxylate plane and the C(1)-C(4) plane is  $20^\circ$ . Many of the aliphatic C-C bond distances (1.460-1.550 Å) are remarkably short. Each piperazinium cation is linked by hydrogen bonds to four different anions with N...O 2.673 and 2.691 Å respectively.

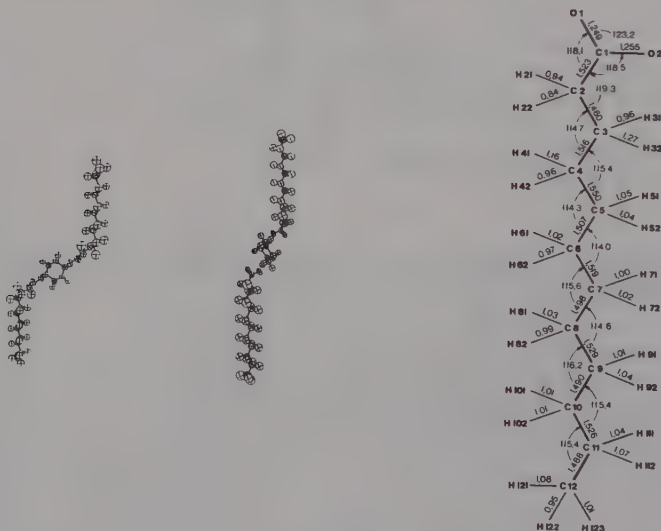


Fig. 1. Piperazinium bis(n-dodecanoate): two views of the piperazinium ion surrounded by two n-dodecanoate anions and bond distances and angles in the n-dodecanoate anion.

# 2,4,6,8-TETRAMETHYL-2,4,6,8-TETRA(2-PYRIDYL)NONANE

$C_{33}H_{40}N_4$

D. AVENEL, D. ADES, R.A. SMITH and T.E. HOGEN-ESCH, 1982. Acta Cryst., B38, 1611-1613.

Monoclinic,  $C2/c$ ,  $a = 20.992$ ,  $b = 6.492$ ,  $c = 21.235$  Å,  $\beta = 101.88^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.069$  for 2025 reflexions.

The molecule has a twofold crystallographic symmetry, with the pyridyl groups distributed alternately on either side of the main chain (Fig. 1). Bond lengths and angles along the chain are normal except for C(1P)-C(3P) 1.572(4), C(3P)-C(9)

1.568(4) Å and C(3)-C(1P)-C(3P) 122.4, C(3P)-C(9)-C(3P') 122.3°.

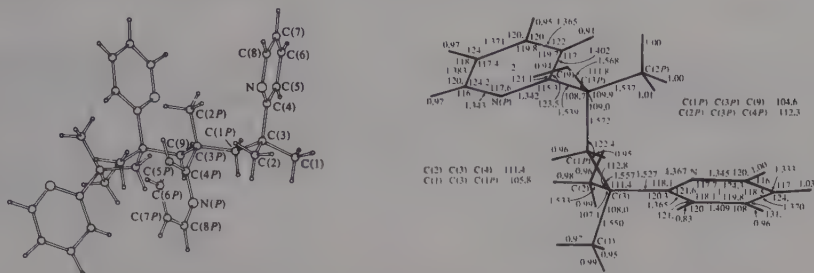


Fig. 1. The  $C_{33}H_{40}N_4$  molecule and details of dimensions of the asymmetric unit.

# BIS(4-METHYLPYRIDINE)HYDROGEN(I) TETRAPHENYLBORATE

$C_{36}H_{35}BN_2$

$(CH_3C_5H_4N)_2H^+, C_{24}H_{20}B^-$

C. GLIDEWELL and H. DIANE HOLDEN, 1982. Acta Cryst., B38, 667-669.

Monoclinic, C2/c,  $a = 16.095$ ,  $b = 10.492$ ,  $c = 16.764$  Å,  $\beta = 108.91^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.079$  for 1004 reflexions.

The cation (Fig. 1) lies across a centre of symmetry with a linear, apparently centrosymmetric N...H...N hydrogen bond; N-H 1.305(7), N...N 2.610(15) Å. The anion lies on a twofold axis. Some dimensions are B(1)-C(11) 1.591(9), B(1)-C(21) 1.560(9) Å and the C-B-C angles are in the range 103.6-115.7°.

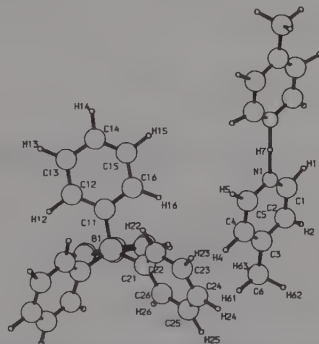


Fig. 1. Two asymmetric units of the structure of bis(4-methylpyridine)hydrogen(I) tetraphenylborate.

# 1,3,5-TRIPHENYL-2,4,6-TRIS(PHENYLIMINO)-1,3,5-HEXAHYDROTRIAZINE (HEXAPHENYLSO-MELAMINE)

$C_{39}H_{30}N_6$  (I)

$(C_6H_5N)_3C_3N_3(C_6H_5)_3$

# 2,4,6-TRIS(DIPHENYLAMINO)-1,3,5-TRIAZINE (HEXAPHENYLMELAMINE)

$C_{39}H_{30}N_6$  (II)

$[(C_6H_5)_2N]_3C_3N_3$

S.V. LINDEMAN, V.E. SHKLOVER, Yu.T. STRUCHKOV, S.N. KUZNETSOV and V.A. PANKRATOV, 1982. Kristallografiya, 27, 65-73 [Sov. Phys. Crystallogr., 27, 36-41].

I. Monoclinic,  $P2_1/c$ ,  $a = 17.309$ ,  $b = 15.188$ ,  $c = 11.831$  Å,  $\beta = 91.07^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.064$  for 2273 reflexions.

II. Trigonal,  $R\bar{3}c$ ,  $a = 14.409$ ,  $c = 25.659$  Å,  $Z = 6$ . Cu radiation,  $R = 0.041$  for 1292 reflexions.

The conformation of the central ring of I is a flattened, twisted boat (Fig. 1). Exocyclic double bonds C=N are elongated to 1.265 Å whilst the endocyclic C-N single bonds are shortened to an average value of 1.398 Å. The molecule II has 32 site symmetry and has a flat central heterocyclic ring (Fig. 1). The endo- and exocyclic C-N bond lengths are respectively 1.337 and 1.374 Å indicating the presence of exocyclic double bonds and a saturated heterocyclic ring as a major resonance form.

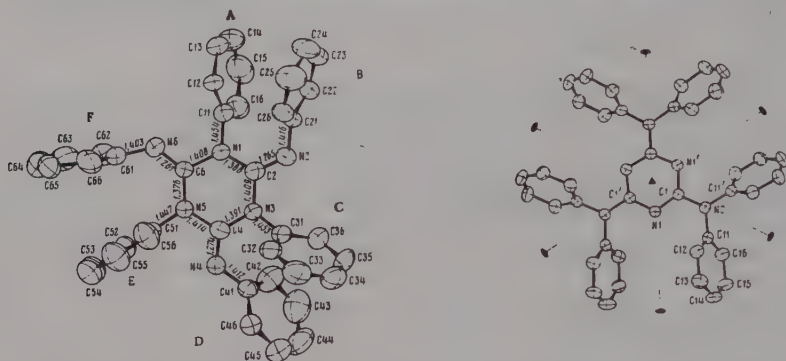


Fig. 1. View of  $C_{39}H_{30}N_6$  (I) (left) with selected distances (Å), and  $C_{39}H_{30}N_6$  (II) (right).

ETHYL  $\alpha$ -(HEXAHYDRO-2-AZEPINYLIDENE)ACETATE  
 $C_{10}H_{17}NO_2$  (I)

ETHYL  $\alpha$ -(TETRAHYDRO-2-PYRROLIDINYLYDENE)ACETATE  
 $C_8H_{13}NO_2$  (II)

M. PHILOCHE-LEVISALLES, C. BOIS, J.-P. CÉLÉRIER and G. LHOMMET, 1982. J. Heterocyclic Chem., 19, 481-484.

I. Monoclinic,  $C2/c$ ,  $a = 19.03$ ,  $b = 5.041$ ,  $c = 21.39$  Å,  $\beta = 93.4^\circ$ ,  $D_m = 1.16$ ,  $Z = 8$ . Mo radiation,  $R = 0.057$  for 1274 reflexions (at  $-30^\circ C$ ).

II. Triclinic,  $P\bar{1}$ ,  $a = 11.20$ ,  $b = 6.833$ ,  $c = 6.201$  Å,  $\alpha = 108.78$ ,  $\beta = 95.97$ ,  $\gamma = 107.15^\circ$ ,  $D_m = 1.19$ ,  $Z = 2$ . Mo radiation,  $R = 0.067$  for 1264 reflexions (at  $-30^\circ C$ ).

The analyses show that the compounds are  $\beta$ -enaminoesters with Z-configuration (Fig. 1). In both compounds, molecules are linked by N-H...O hydrogen bonds, 2.578-2.931 Å.

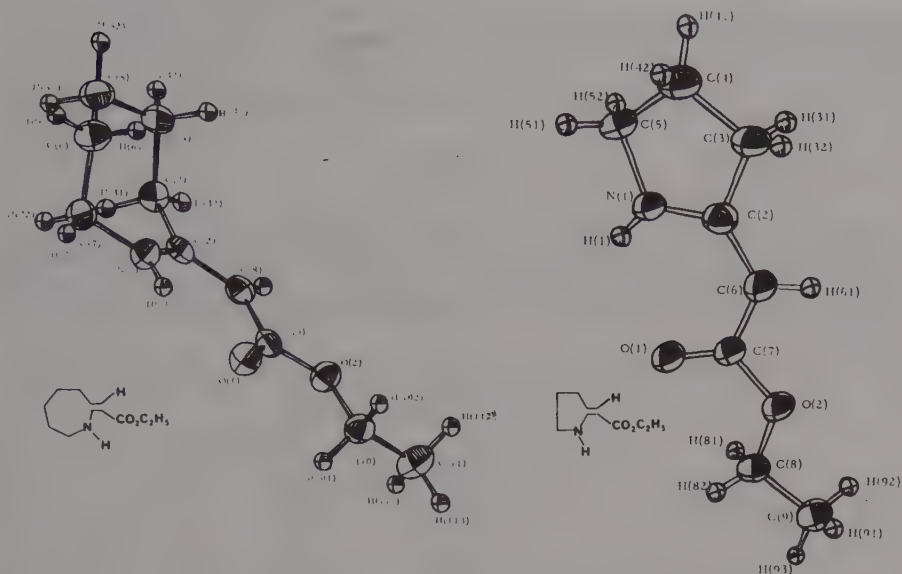


Fig. 1. Views of C<sub>10</sub>H<sub>17</sub>NO<sub>2</sub> (I) (left) and C<sub>8</sub>H<sub>13</sub>NO<sub>2</sub> (II) (right).

1,10-DIAZA-4,7,13,16-TETRAAZONIUMCYCLOOCTADECANE DINITRATE DICHLORIDE DIHYDRATE  
 C<sub>12</sub>H<sub>34</sub>Cl<sub>2</sub>N<sub>8</sub>O<sub>6</sub>·2(H<sub>2</sub>O) (C<sub>12</sub>H<sub>34</sub>N<sub>6</sub>)(NO<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub>·2(H<sub>2</sub>O)

J. CULLINANE, R.I. GELB, T.N. MARGULIS and L.J. ZOMPA, 1982. J. Am. Chem. Soc., **104**, 3048-3053.

Monoclinic, P2<sub>1</sub>/a, a = 11.569, b = 17.186, c = 5.869 Å, β = 94.74°, Z = 2. Cu radiation, R = 0.039 for 1791 reflexions.

The structure (Fig. 1) consists of protonated macrocycles whose 7NH<sub>2</sub><sup>+</sup> groups are H-bonded to Cl<sup>-</sup> anions and H<sub>2</sub>O molecules, the latter being H-bonded to NO<sub>3</sub><sup>-</sup> anions. The N...Cl hydrogen-bonded distances are 3.07-3.28 Å, other dimensions are normal.

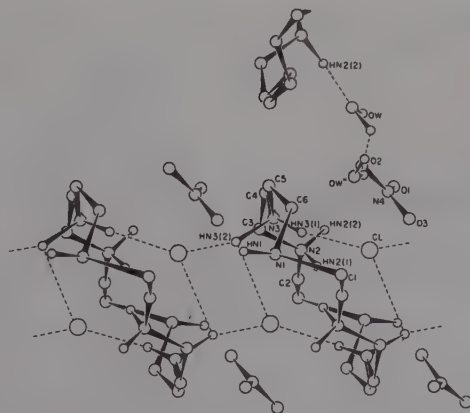
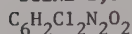


Fig. 1. The structure of (C<sub>12</sub>H<sub>34</sub>N<sub>6</sub>)(NO<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub>·2(H<sub>2</sub>O) viewed along the a axis.

9,10-DIOXA-syn-(HYDRO,CHLORO)BIMANE (3,7-DICHLORO-1,5-DIAZABICYCLO[3.3.0]OCTA-3,6-DIENE-2,8-DIONE)



I. GOLDBERG and E.M. KOSOWER, 1982. *J. Phys. Chem.*, **86**, 332-335.

Triclinic,  $P\bar{1}$ ,  $a = 6.630$ ,  $b = 7.192$ ,  $c = 9.264$  Å,  $\alpha = 75.54^\circ$ ,  $\beta = 110.92^\circ$ ,  $\gamma = 117.47^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.031$  for 1499 reflexions.

The crystal structure consists of layers of planar molecules approximately perpendicular to the  $b$  axis, adjacent layers being related to each other by inversion. Within each layer, the molecules (Fig. 1) associate mainly through C-H...O=C bonds (along a direction parallel to  $a$ ) as well as through C-Cl...C-Cl dipolar interactions (along directions parallel to  $c$  and  $a+c$ ). Every molecule is thus in contact with six neighbours:

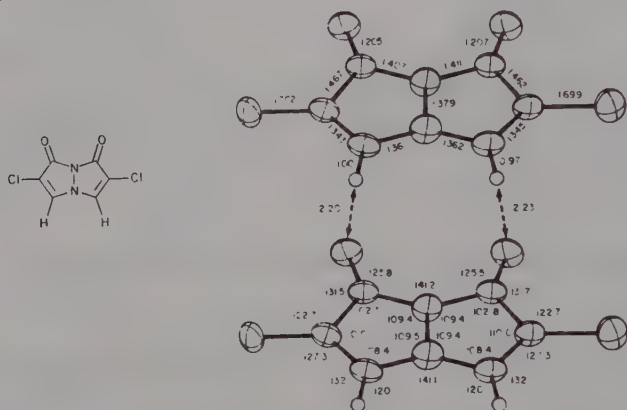
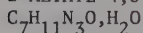


Fig. 1. The bimane molecule and a drawing of two molecules showing the C-H...O hydrogen bonds.

2-METHYL-4,5,6,7-TETRAHYDOPYRAZOLO[3,4-c]PYRIDIN-3-OL MONOHYDRATE



L. BREHM, 1982. *Acta Cryst.*, **B38**, 2741-2744.

Monoclinic,  $P2_1/c$ ,  $a = 8.204$ ,  $b = 7.352$ ,  $c = 15.320$  Å,  $\beta = 118.52^\circ$ ,  $D_m = 1.40$ ,  $Z = 4$ . Mo radiation,  $R = 0.044$  for 1309 reflexions.

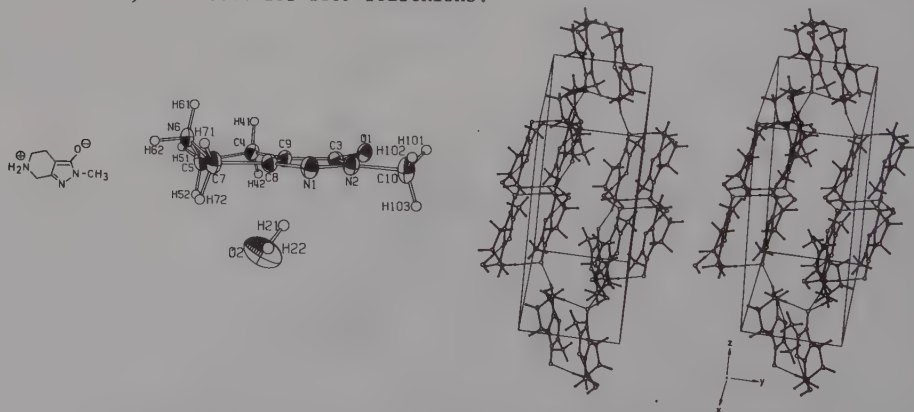


Fig. 1.  $\text{C}_7\text{H}_{11}\text{N}_3\text{O}_2\cdot\text{H}_2\text{O}$ : a view of the molecule and a stereoscopic view of the molecular packing.

The molecule (Fig. 1) is a zwitterion in which bond lengths and angles are in good agreement with the expected values. The molecules are linked in the crystal by hydrogen bonds with N...O 2.832(2) and 2.716(3), O(water)...N 2.922(4) and O(water)...O 2.780(3) Å. All other intermolecular contacts correspond to van der Waals interactions.

## 3H-5-HYDROXYINDOLE



G. BOCELLI and M.F. GRENIER-LOUSTALOT, 1982. *J. Mol. Struct.*, **96**, 127-131.

Monoclinic,  $P2_1/c$ ,  $a = 5.836$ ,  $b = 15.164$ ,  $c = 7.507$  Å,  $\beta = 84.2^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.054$  for 1249 reflexions.

The molecule (Fig. 1) is approximately planar (maximum deviation 0.8 Å), with N-C4 1.413(2), N-C8 1.341(3), C8-O2 1.241(2), C1-O1 1.367(2) Å.

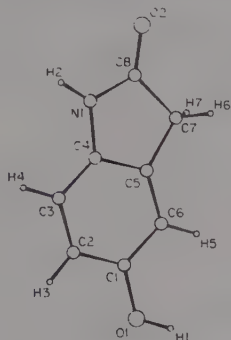
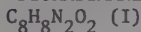
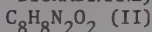


Fig. 1. 3H-5-Hydroxyindole.

2,8-DIMETHYL-1,5-DIAZABICYCLO[3.3.0]OCTA-2,7-DIENE-4,6-DIONE (syn-(CH<sub>3</sub>,H)-9,10-DIOXABIMANE)



2,6-DIMETHYL-1,5-DIAZABICYCLO[3.3.0]OCTA-2,6-DIENE-4,8-DIONE (anti-(CH<sub>3</sub>,H)-9,10-DIOXABIMANE)



2,6-DIMETHYL-3,5-DIOXO-4,11-DIAZATRICYCLO[5.3.1.0<sup>4,11</sup>]UNDECA-1,6-DIENE-9,9-DICARBONITRILE ( $\mu$ -[C(CN)<sub>2</sub>]-syn(CH<sub>2</sub>,CH<sub>3</sub>)-9,10-DIOXABIMANE)



I. GOLDBERG, J. BERNSTEIN and E.M. KOSOWER, 1982. *Acta Cryst.*, **B38**, 1990-2000.

I. Orthorhombic,  $Pnma$ ,  $a = 13.987$ ,  $b = 6.651$ ,  $c = 8.398$  Å,  $Z = 4$ . Mo radiation,  $R = 0.046$  for 960 reflexions ( $a = 13.970$ ,  $b = 6.547$ ,  $c = 8.372$  Å at 193 K).

II. Triclinic,  $P\bar{1}$ ,  $a = 6.374$ ,  $b = 7.595$ ,  $c = 8.442$  Å,  $\alpha = 101.24$ ,  $\beta = 95.51$ ,  $\gamma = 92.44^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.053$  for 1426 reflexions.

III. Monoclinic,  $P2_1/c$ ,  $a = 9.975$ ,  $b' = 7.413$ ,  $c = 17.008$  Å,  $\beta = 100.10^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.047$  for 1355 reflexions.

In (I) (Fig. 1) the molecules have all non-hydrogen atoms (and four hydrogens) located on mirror planes perpendicular to  $b$  producing a layered structure; within each layer symmetry-equivalent molecules are almost at right angles to each other and form infinite stacks of almost entirely overlapping species with 3.273 Å



separating successive molecular planes. The two independent molecules in (II) (Fig. 1) lie on inversion centres and are almost exactly parallel. They are planar to within 0.01 Å the molecular plane of one being rotated 57° with respect to that of the other. Sheets of molecules are formed parallel to the  $1\bar{1}2$  plane. The molecules in (III) (Fig. 1) are bent about the N-N bond - the dihedral angle between the mean planes of the two five-membered rings is 139.3° and the C(4)-N(5)-C(6) angle is reduced to 121.4(2)°; C(4) and C(6) are 0.06 and 0.08 Å respectively from the mean ring planes and the sum of the angles about N is 338°, N(5) and 343°, N(1). The N-N distances in I, II and III are 1.379(2), 1.368(4) and 1.372(4), 1.394(3) Å; N-C(=C) is 1.376-1.377 in I and II and 1.406 Å in III and N-C(=O) is 1.396 (II), 1.411 (I), 1.435 Å (III). In (III) C(13)-C(15) is 1.569(4) and C(14)-C(15) 1.565(5) Å. All three structures contain short C-H...O contacts in the range 3.18-3.35 Å (C...O) and 2.37-2.63 Å (H...O) with C-H...O angles in the range 130-175°.

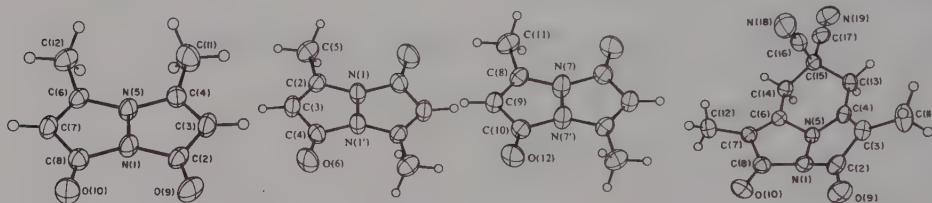


Fig. 1. Views of syn- $C_8H_8N_2O_2$  (left), two independent anti- $C_8H_8N_2O_2$  (centre) and  $C_{13}H_{10}N_4O_2$  (right).

5-METHYL-1,3,4,6-TETRAOXOPERHYDROPYRROLO[3,4-c]PYRIDINE  
 $C_8H_8N_2O_4$

A. AMORESE, E. GAVUZZO, F. MAZZA, G. CASINI and M. FERAPPI, 1982. *Acta Cryst.*, B38, 3145-3147.

Monoclinic,  $P2_1/c$ ,  $a = 11.227$ ,  $b = 6.177$ ,  $c = 12.828$  Å,  $\beta = 111.68^\circ$ ,  $D_m = 1.57$ ,  $Z = 4$ . Mo radiation,  $R = 0.064$  for 1501 reflexions.

The two rings are cis-fused (Fig. 1); the five-membered ring is in envelope conformation and the six-membered ring has sofa form.

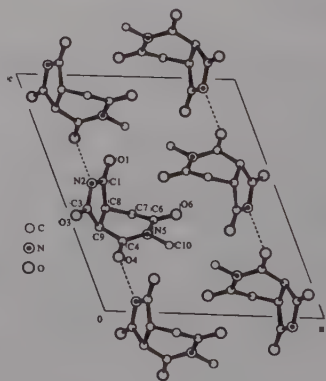
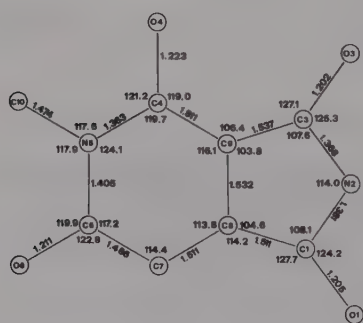
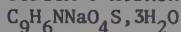


Fig. 1. Dimensions and packing for  $C_8H_8N_2O_4$ .

## SODIUM 8-HYDROXYQUINOLINE-5-SULPHONATE TRIHYDRATE



B. VIOSSAT, P. KHODADAD and N. RODIER, 1982. Bull. Soc. Chim. Fr., I289-I291.

Triclinic,  $P\bar{1}$ ,  $a = 12.438$ ,  $b = 8.688$ ,  $c = 7.561$  Å,  $\alpha = 112.56^\circ$ ,  $\beta = 95.14^\circ$ ,  $\gamma = 118.06^\circ$ ,  $D_m = 1.55$ ,  $Z = 2$ . Mo radiation,  $R = 0.039$  for 2331 reflexions.

The structure consists of layers of hydroxyquinoline sulphonate groups parallel to the (100) plane. The layers are linked together by interactions between the  $\text{Na}^+$  ion and O or N atoms and hydrogen bonds between water molecules and sulphonate groups and hydroxyl groups. In the same layer, cohesion is due to van der Waals interactions. The  $\text{Na}^+$  ion has a strongly distorted octahedral environment with  $\text{Na} \cdots \text{O} \ 2.376\text{--}2.402(3)$  Å,  $\text{Na} \cdots \text{N} \ 2.445(3)$  Å.

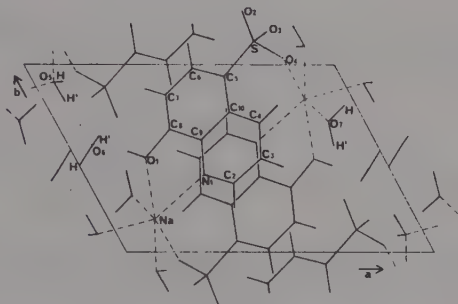
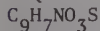


Fig. 1. The projection of  $\text{C}_9\text{H}_6\text{NNaO}_4\text{S} \cdot 3\text{H}_2\text{O}$  onto the (001) plane.

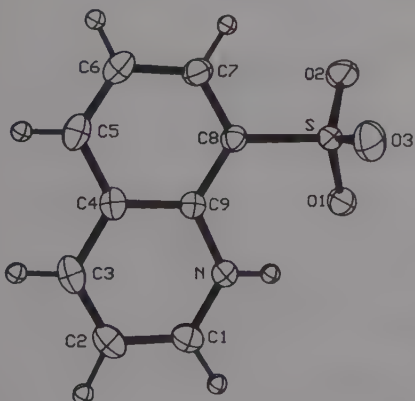
## 8-QUINOLINESULPHONIC ACID



B.M. GATEHOUSE, 1982. Cryst. Struct. Comm., 11, 489-492.

Monoclinic,  $P2_1/c$ ,  $a = 7.147$ ,  $b = 16.258$ ,  $c = 8.068$  Å,  $\beta = 115.38^\circ$ ,  $D_m = 1.62$ ,  $Z = 4$ . Mo radiation,  $R = 0.038$  for 1715 reflexions.

A view of this monoclinic form is shown in Fig. 1 with bond lengths.



$\text{C}(1) - \text{C}(2)$	$1.392(5)$ Å	$\text{C}(6) - \text{C}(7)$	$1.413(4)$
$\text{C}(2) - \text{C}(3)$	$1.361(5)$	$\text{C}(7) - \text{C}(8)$	$1.375(5)$
$\text{C}(3) - \text{C}(4)$	$1.415(5)$	$\text{C}(8) - \text{C}(9)$	$1.412(4)$
$\text{C}(4) - \text{C}(5)$	$1.415(5)$	$\text{C}(8) - \text{S}$	$1.785(3)$
$\text{C}(4) - \text{C}(9)$	$1.422(4)$	$\text{C}(9) - \text{N}$	$1.376(4)$
$\text{C}(5) - \text{C}(6)$	$1.360(6)$	$\text{C}(1) - \text{N}$	$1.325(4)$
$\text{S} - \text{O}(1)$	$1.456(3)$		
$\text{S} - \text{O}(2)$	$1.437(4)$		
$\text{S} - \text{O}(3)$	$1.448(4)$		

Fig. 1. The monoclinic form of  $\text{C}_9\text{H}_7\text{NO}_3\text{S}$ .

## 8-QUINOLINESULPHONIC ACID



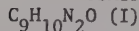
B.M. GATEHOUSE, 1982. Cryst. Struct. Comm., **11**, 493-495.

Triclinic,  $\text{P}\bar{1}$ ,  $a = 7.893$ ,  $b = 8.028$ ,  $c = 10.228 \text{ \AA}$ ,  $\alpha = 92.61$ ,  $\beta = 131.84$ ,  $\gamma = 107.68^\circ$ ,  $D_m = 1.62$ ,  $Z = 2$ . Mo radiation,  $R = 0.035$  for 1609 reflexions.

For details of the monoclinic form, see (1). Bond lengths in the two forms are the same within the accuracy of the determinations. The differences between the two forms are found in the packing of the molecules.

# 1. Preceding Report.

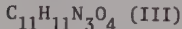
## 5-ETHOXYIMIDAZO[1,2-a]PYRIDINE



## ETHYL 8-METHYLIMIDAZO[1,2-a]PYRIDINE-2-CARBOXYLATE



## ETHYL 6-METHYL-3-NITROIMIDAZO[1,2-a]PYRIDINE-2-CARBOXYLATE



J.C. TEULADE, R. ESCALE, J.C. ROSSI, J.P. CHAPAT, G. GRASSY and M. PAYARD, 1982. Aust. J. Chem., **35**, 1761-1768.

I. Monoclinic,  $\text{Cc}$ ,  $a = 7.392$ ,  $b = 16.866$ ,  $c = 7.702 \text{ \AA}$ ,  $\beta = 117.94^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.048$  for 360 reflexions.

II. Monoclinic,  $\text{P2}_1/\text{a}$ ,  $a = 12.434$ ,  $b = 11.358$ ,  $c = 7.472 \text{ \AA}$ ,  $\beta = 91.54^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.048$  for 1024 reflexions.

III. Triclinic,  $\text{P1}$ ,  $a = 11.641$ ,  $b = 8.361$ ,  $c = 6.191 \text{ \AA}$ ,  $\alpha = 83.64$ ,  $\beta = 101.64$ ,  $\gamma = 88.09^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.057$  for 798 reflexions.

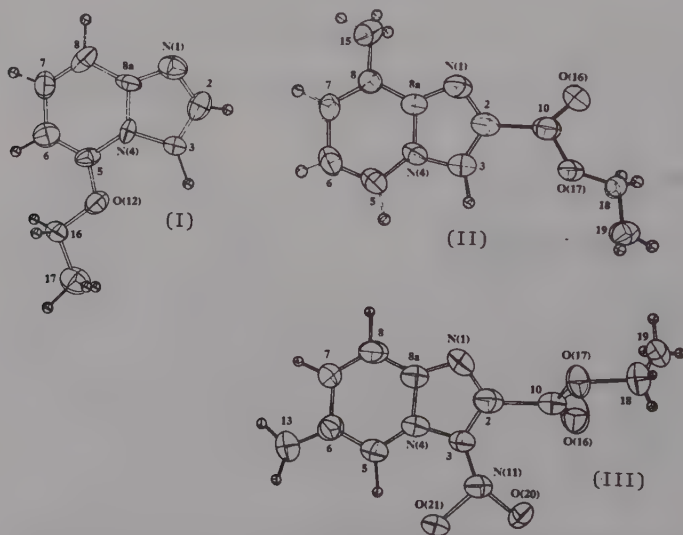


Fig. 1. Perspective views of the [1,2-a]pyridine derivatives (I), (II), (III).

The structures of the three molecules were confirmed as those shown in Fig. 1. Bond lengths have expected values with pronounced double-bond character of the 5-6 and 7-8 bonds (I: 1.34(2), 1.34(2); II: 1.328(6), 1.352(5); III: 1.37(1), 1.37(1) Å). The fused ring systems are planar within experimental error.

### 3-METHOXY-2-METHYL-8-METHYLAMINOIMIDAZO[1,2-b]PYRIDAZINE

$C_9H_{12}N_4O$

G.B. BARLIN, I.L. BROWN, L. GOLIC and V. KAUCIC, 1982. Aust. J. Chem., **35**, 423-430.

Triclinic,  $P\bar{1}$ ,  $a = 8.090$ ,  $b = 11.357$ ,  $c = 11.618$  Å,  $\alpha = 88.81^\circ$ ,  $\beta = 70.27^\circ$ ,  $\gamma = 89.47^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.050$  for 1687 reflexions.

The fused imidazo[1,2-b]pyridazine ring system (Fig. 1) is nearly planar, with the largest deviations being C(8a) 0.019 and C(6') 0.013 Å. The dihedral angle between the two rings is  $1.2(7)^\circ$ . Bond lengths and angles have expected values.

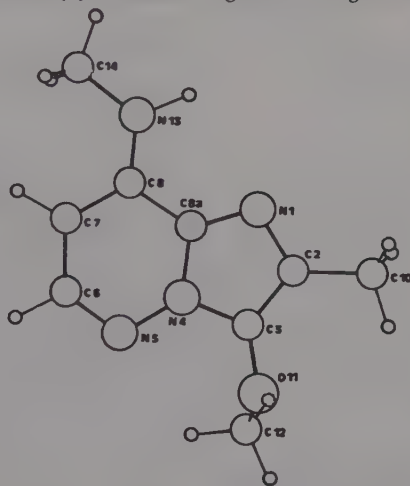


Fig. 1. Perspective view of 3-methoxy-2-methyl-8-methylaminoimidazo[1,2-b]-pyridazine.

### 7-CHLORO-8-METHYLCARBOSTYRIL

$C_{10}H_8ClNO$

R. RADHAKRISHNAN and S. RAGHUNATHAN, 1982. Acta Cryst., **B38**, 326-327.

Monoclinic,  $P2_1/c$ ,  $a = 14.343$ ,  $b = 4.175$ ,  $c = 16.023$  Å,  $\beta = 114.963^\circ$ ,  $D_m = 1.469$ ,  $Z = 4$ . Cu radiation,  $R = 0.052$  for 1388 reflexions.

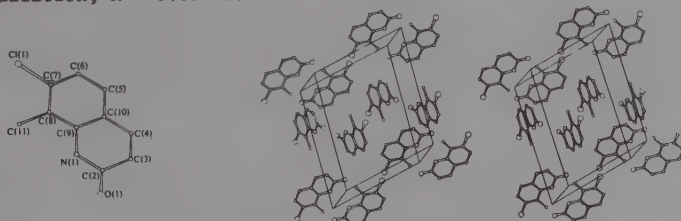


Fig. 1. A perspective view of the  $C_{10}H_8ClNO$  molecule (left) and a stereoscopic view of the molecular packing (right).

The entire molecule (Fig. 1) is nearly planar and has bond lengths and angles close to normal values. The molecules are linked in the crystal by N-H...O hydrogen bonds with N...O 2.878 Å.

SODIUM 8-METHYLTHIOQUINOLINE-5-SULPHONATE HEMIHYDRATE  
 $C_{10}H_8NNaO_3S_2 \cdot 0.5H_2O$

A.D. OZOLA, Ya.K. OZOLS, A.A. KEMME, Ya.Ya. BLEIDELIS and Ya.V. ASHAKS, 1982. Zh. Strukt. Khim., 23-6, 98-102 [J. Struct. Chem., 23, 905-908].

Monoclinic, Aa,  $a = 10.889$ ,  $b = 5.510$ ,  $c = 39.752$  Å,  $\beta = 105.10^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.046$  for 1815 reflexions.

The geometry of the two independent anions is shown in Fig. 1. The quinoline rings are planar within 0.02 Å. The  $Na^+$  cations are each coordinated to six O atoms in distorted octahedral geometry with Na-O 2.35-2.85 Å.

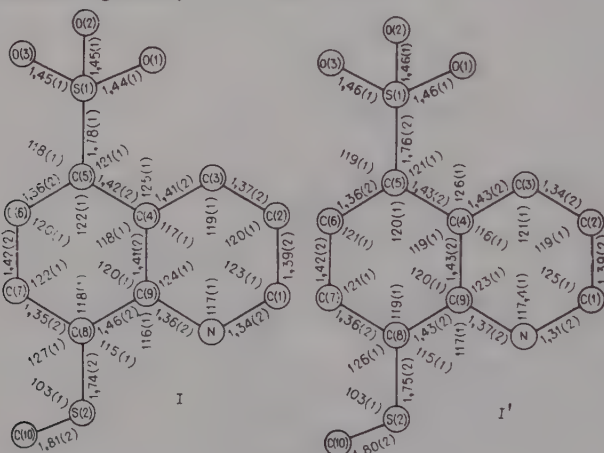


Fig. 1. Geometry of the two independent  $C_{10}H_8NO_3S_2^-$  anions.

N-METHYL-8-FLUOROQUINOLINIUM CHLORIDE HYDRATE  
 $C_{10}H_9ClFN, H_2O$

S.R. WALTER, M. BARFIELD, G.W. GRIBBLE and K.W. HADEN, 1982. J. Cryst. Spectrosc. Res., 12, 473-480.

Monoclinic,  $P2_1/n$ ,  $a = 6.938$ ,  $b = 7.855$ ,  $c = 18.592$  Å,  $\beta = 94.72^\circ$ ,  $D_m = 1.415$ ,  $Z = 4$ . Mo radiation,  $R = 0.055$  for 1040 reflexions.

Steric interactions between the fluorine and the methyl group are evident from the structural data, but the planarity of the quinolinium ring is not significantly distorted (Fig. 1). The F atom is in the quinoline plane and C(9) is 0.085 Å out of this plane. There is significant angle bending (F-C(8)-C(7)  $115.2(4)^\circ$ , F-C(8)-C(8A)  $120.83^\circ$ , C(2)-N-C(9)  $118.4(3)^\circ$ , C(8A)-N-C(9)  $121.3(3)^\circ$ ).

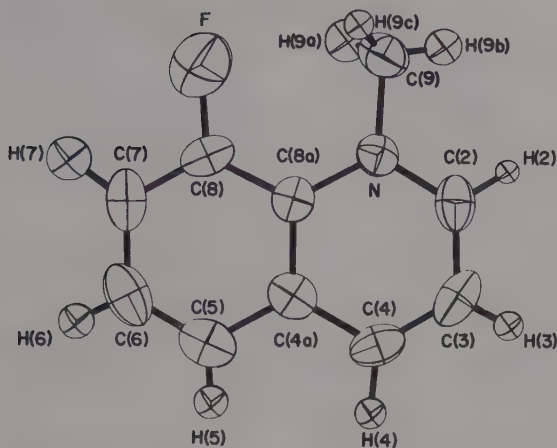


Fig. 1. The  $C_{10}H_9FN$  cation.

### (3-INDOLYL)ACETIC ACID

$C_{10}H_9NO_2$

K. CHANDRASEKHAR and S. RAGHUNATHAN, 1982. *Acta Cryst.*, **B38**, 2534-2535.

Monoclinic,  $P2_1/c$ ,  $a = 17.938$ ,  $b = 5.254$ ,  $c = 9.591$  Å,  $\beta = 106.34^\circ$ ,  $D_m = 1.37$ ,  $Z = 4$ . Co radiation,  $R = 0.045$  for 921 reflexions.

This analysis is a re-investigation of a previously determined structure (1). Bond lengths and angles are generally as expected. The angle between the indole ring and carboxyl-group plane is  $89.9(4)^\circ$  cf.  $60.9^\circ$  (1). The molecules (Fig. 1) are linked in the crystal into dimers by centrosymmetric O-H...O hydrogen bonds, with  $O \cdots O$  2.653(4) Å.

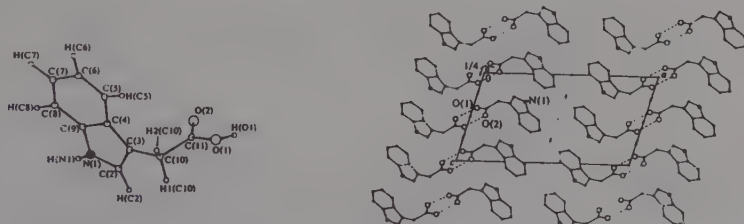


Fig. 1.  $C_{10}H_9NO_2$ : perspective view of the molecule and a view of the crystal structure projected down  $b$ ; hydrogen bonds are shown by broken lines.

1. *Structure Reports*, **29**, 726.

### 3,4-DIHYDRO-2,5-DIMETHYL-7-PROPYLIMIDAZO[5,1-f][1,2,4]TRIAZINE

$C_{10}H_{16}N_4$

P. MURRAY-RUST, J. MURRAY-RUST, D.I.C. SCOPES and A.W. OXFORD, 1982. *Acta Cryst.*, **B38**, 2694-2695.

Monoclinic,  $P2_1/c$ ,  $a = 7.90$ ,  $b = 11.83$ ,  $c = 14.23$  Å,  $\beta = 56.75^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.068$  for 1171 reflexions.



In the molecule (Fig. 1) the propyl group has a perpendicular-trans conformation, very similar to that found in related molecules. The molecules are linked in the crystal by hydrogen bonds (N(3)...N(6') 2.94(1) Å).

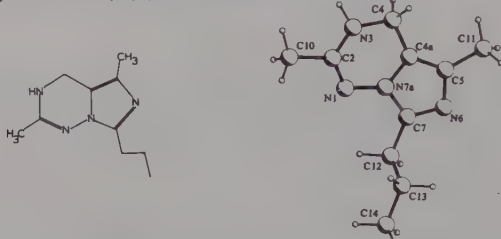
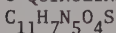


Fig. 1.  $C_{10}H_{16}N_4$ : perspective view of the molecule.

8-QUINOLINESULPHONE(3-NITRO-1,2,4-TRIAZOLIDE)



J. ENGELS, U. KRAHMER, L. ZSOLNAI and G. HUTTNER, 1982. Justus Liebigs Ann. Chem., 745-753.

Triclinic,  $P\bar{1}$ ,  $a = 7.947$ ,  $b = 8.283$ ,  $c = 9.810$  Å,  $\alpha = 97.1$ ,  $\beta = 95.5$ ,  $\gamma = 99.2^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.064$  for 1099 reflexions (at 233 K).

In the molecule (Fig. 1) the triazole ring and the quinoline ring system are nearly eclipsed (N(1)S(1)-C(3)C(4)  $-65.0(4)^\circ$ ), and the S-O(2) bond lies almost in the quinoline plane (O(2)S(1)-C(3)C(11)  $5.1(4)^\circ$ ). Bond lengths and angles have normal values.

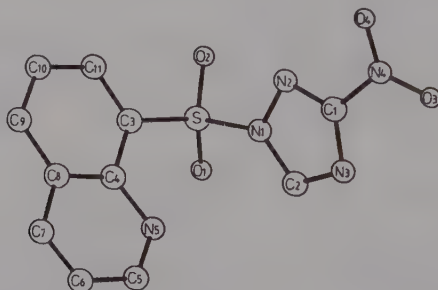
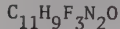


Fig. 1. Perspective view of  $C_{11}H_7N_5O_4S$ .

1,2-DIMETHYL-5-TRIFLUOROACETYL-2H-CYCLOPENTA[d]PYRIDAZINE



R.E. STENKAMP and R.P. Ko, 1982. Acta Cryst., B38, 994-996.

Orthorhombic,  $Pnma$ ,  $a = 18.059$ ,  $b = 6.977$ ,  $c = 8.338$  Å,  $Z = 4$ . Cu radiation,  $R = 0.056$  for 840 reflexions.

The largely planar molecules pack in this crystal form with their molecular mirror planes coincident with the crystallographic mirror. The bond lengths (Fig. 1) indicate substantial mixing of single- and double-bond character throughout the heterocyclic ring system. The N-N distance in the six-membered ring, 1.369 Å, is consistent with substantial single-bond character in the N-N bond.

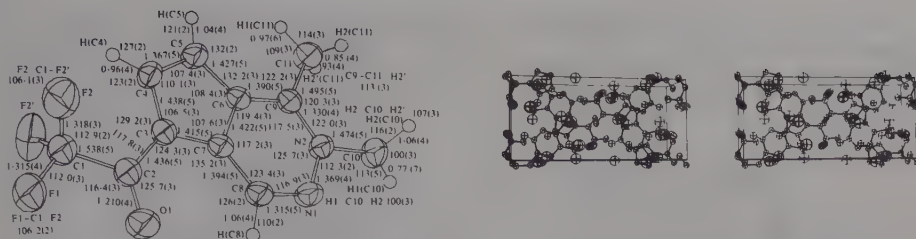


Fig. 1.  $C_{11}H_9F_3N_2O$ : bond lengths and angles (left) and a stereoscopic view of the unit cell contents (right).

4-(2-AMINO-4-OXO-2-IMIDAZOLIN-5-YLIDENE)-2-BROMO-4,5,6,7-TETRAHYDROPYRROLO[2,3-c]-AZEPIN-8-ONE METHANOL SOLVATE

$C_{11}H_{10}BrN_5O_2 \cdot CH_4O$

C.A. MATTIA, L. MAZZARELLA and R. PULITI, 1982. Acta Cryst., B38, 2513-2515.

Monoclinic,  $P2_1/n$ ,  $a = 11.943$ ,  $b = 16.252$ ,  $c = 7.253$  Å,  $\beta = 93.43^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.042$  for 1997 reflexions.

The molecule (Fig. 1) contains a seven-membered ring in a distorted boat conformation with an approximate mirror plane passing through C(6) and the mid-point of the C(3)-C(4) bond. The pyrrole ring is planar whereas in the imidazole ring C(9) is slightly displaced out of the best plane through the remaining atoms. The molecules are held together in the crystal by N-H...O and O-H...O hydrogen bonds which involve the methanolic O atom.

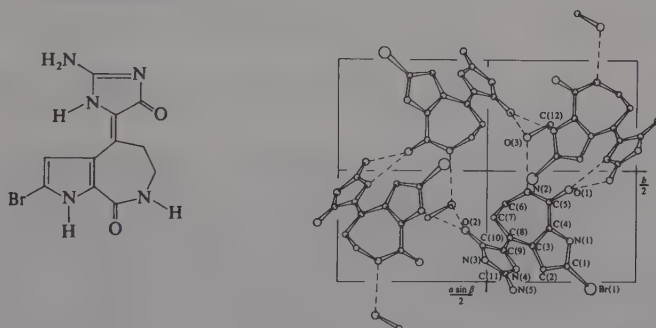


Fig. 1.  $C_{11}H_{10}BrN_5O_2 \cdot CH_4O$ : molecular structure and packing arrangement in the crystal; hydrogen bonds are shown as dashed lines.

1,4-DIACETYL-1,4-DIHYDROQUINOXALINE  
 $C_{12}H_{12}N_2O_2$  (I)

1,2-DIACETYL-1,2-DIHYDROBENZO[c]CINNOLINE  
 $C_{16}H_{14}N_2O_2$  (II)

J. ARMAND and M. PHILOCHE-LEVISALLES, 1982. Canad. J. Chem., 60, 2792-2796.

I. Orthorhombic,  $Pnma$ ,  $a = 10.438$ ,  $b = 15.363$ ,  $c = 6.638$  Å,  $D_m = 1.32$ ,  $Z = 4$ . Mo radiation,  $R = 0.047$  for 909 reflexions.

II. Orthorhombic,  $Pbcn$ ,  $a = 19.350$ ,  $b = 9.797$ ,  $c = 14.110$  Å,  $D_m = 1.30$ ,  $Z = 8$ . Mo radiation,  $R = 0.051$  for 1714 reflexions.

Molecule I has crystallographic mirror symmetry, with the mirror plane perpendicular to the N1...N4 axis (Fig. 1). The 1,4-dihydropyrazine ring has a boat conformation. In II, the dihedral angle between the planar phenyl rings is  $19.5^\circ$  and the heterocyclic ring is not planar as a result.

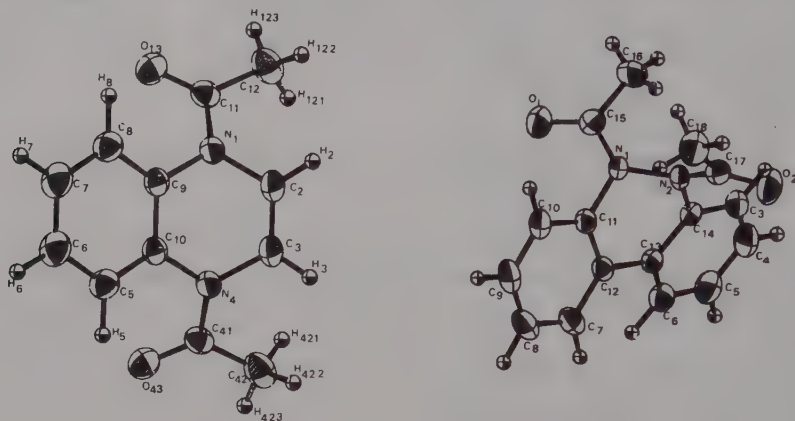


Fig. 1. Views of  $C_{12}H_{22}N_2O_2$  (left) and  $C_{16}H_{14}N_2O_2$  (right).

4,6-BIS(METHOXYCARBONYL)-3,7-DIMETHYL-1,5-DIAZABICYCLO[3.3.0]OCTA-3,6-DIENE-2,8-DIONE  
 $C_{12}H_{12}N_2O_6$

E.M. KOSOWER, D. FAUST, M. BEN-SHOSHAN and I. GOLDBERG, 1982. *J. Org. Chem.*, **47**, 214-221.

Monoclinic,  $P2_1/c$ ,  $a = 9.668$ ,  $b = 18.009$ ,  $c = 7.985$  Å,  $\beta = 112.77^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.059$  for 1322 reflexions.

The strong fluorescence exhibited by this and all related bimanes suggested that these molecules have a common syn arrangement. This was confirmed by X-ray analysis (Fig. 1). The biman system is non-planar with a dihedral angle of  $161.2^\circ$  between the mean planes of the two five-membered rings. This conformation is intermediate between values (ca.  $180$  and  $140^\circ$ ) previously reported for bimanes (1), and the deviation from planarity here may result from the intramolecular  $O \cdots C=O$  interaction between the two ester groups. The  $O \cdots C$  distance is  $2.728$  Å and the  $O \cdots C=O$  angle  $84.4^\circ$ . Both carboxymethoxy groups are rotated out of the adjacent ring planes. Bond dimensions resemble those in the other bimanes and reflect some  $\pi$  electron delocalisation.

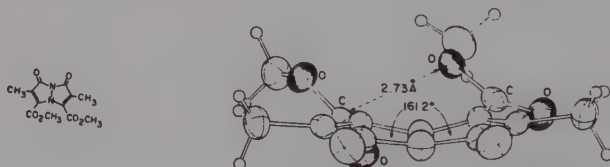


Fig. 1. Formula and structure of the biman  $C_{12}H_{12}N_2O_6$ .

1. *Structure Reports*, **46B**, 257, 398.

3,4-DIHYDRO-2,4,4,5,7-PENTAMETHYLPYRROLO[1,2-b]PYRIDAZINE  
 $C_{12}H_{18}N_2$

D.E. JACKMAN, A.W. BURGSTAHLER, B. LEE and R.I. SHELTON, 1982. Cryst. Struct. Comm., 11, 1111-1114.

Monoclinic,  $P2_1/c$ ,  $a = 8.200$ ;  $b = 10.639$ ,  $c = 13.565$  Å,  $\beta = 104.39^\circ$ ,  $D_m = 1.11$ ,  $Z = 4$ . Mo radiation,  $R = 0.0502$  for 1245 reflexions.

The analysis established the structure shown in Fig. 1. Atoms C2, C4a, C5, C6, C7 and N8 are trigonal with the sum of the bond angles at each of these atoms deviating from  $360^\circ$  by no more than  $0.2^\circ$ . The pyrrole ring comprising atoms C4a, C5, C6, C7 and N8 is planar with the rms and the maximum deviation of the five atoms in this group from their mean plane being 0.005 and 0.006 Å respectively. Atoms C3 and C4 lie on opposite sides of the mean plane of the pyridazine ring by 0.26 Å.

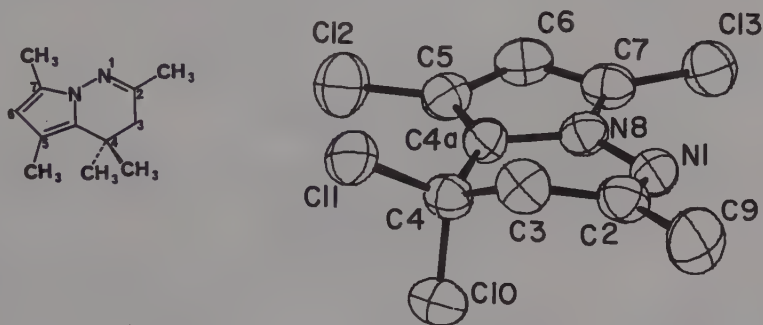


Fig. 1. A view of  $C_{12}H_{18}N_2$ .

6,7-DIMETHOXY-2,3-DIMETHYL-1(2H)-ISOQUINOLINONE  
 $C_{13}H_{15}NO_3$

M.P. GUPTA and K. BANERJEE, 1982. Acta Cryst., B38, 2947-2948.

Monoclinic,  $P2_1/c$ ,  $a = 10.663$ ,  $b = 13.809$ ,  $c = 8.206$  Å,  $\beta = 110.95^\circ$ ,  $D_m = 1.36$ ,  $Z = 4$ . Mo radiation,  $R = 0.080$  for 1983 reflexions.

The molecule (Fig. 1) has a nearly planar quinoline system in which the two six-membered rings are inclined at  $2.1(3)^\circ$ . Bond lengths and angles are generally as expected. The molecules lie in the crystal in layers nearly parallel to the (010) plane.

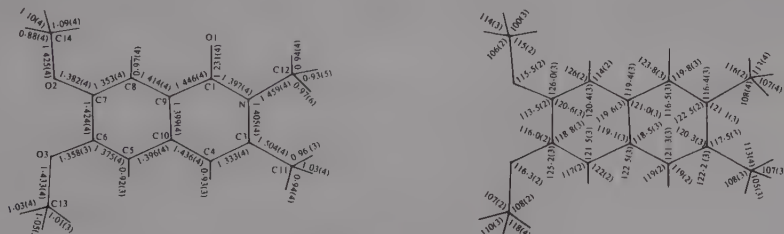


Fig. 1.  $C_{13}H_{15}NO_3$ : bond lengths and angles.

2,2-DIMETHYL-3-(1-METHYLETHYLIDENE)AMINO-4-OXO-1,2,3,4-TETRAHYDROQUINAZOLINE  
 $C_{13}H_{17}N_3O$

C. PELIZZI, G. PELIZZI and G. PREDIERI, 1982. *Gazz. Chim. Ital.*, **112**, 343-344.

Monoclinic,  $P2_1/n$ ,  $a = 9.061$ ,  $b = 14.828$ ,  $c = 9.445$  Å,  $\beta = 88.68^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.0483$  for 1359 reflexions.

The analysis establishes the structure shown in Fig. 1. The two six-membered rings are mutually inclined at  $3.4^\circ$ . The benzene ring is planar within 0.002 Å with C-C distances ranging from 1.356(4) to 1.390(4) Å. The puckered pyrimidine ring adopts a sofa conformation with five atoms practically coplanar, the sixth atom (C(7)) being 0.59 Å out of this plane. Molecules are linked by N(1)-H...O hydrogen bonds (2.880(3) Å).

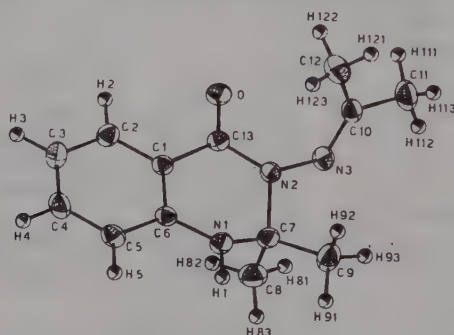


Fig. 1. A view of  $C_{13}H_{17}N_3O$ .

2,6-BIS(METHOXYCARBONYL)-4,4,8,8-TETRAKIS(TRIFLUOROMETHYL)-1,5-DIAZABICYCLO[3.3.0]-OCTANE (cis FORM)  
 $C_{14}H_{12}F_{12}N_2O_4$  (I)

2,6-BIS(ETHOXYCARBONYL)-4,4,8,8-TETRAKIS(TRIFLUOROMETHYL)-1,5-DIAZABICYCLO[3.3.0]-OCTANE (trans FORM)  
 $C_{16}H_{16}F_{12}N_2O_4$  (II)

2,6-BIS(ETHOXYCARBONYL)-4,4,8,8-TETRAKIS(TRIFLUOROMETHYL)-1,5-DIAZABICYCLO[3.3.0]-OCTANE (cis FORM)  
 $C_{16}H_{16}F_{12}N_2O_4$  (III)

K. BURGER, H. SCHICKANEDER, F. HEIN, A. GIEREN, V. LAMM and H. ENGELHARDT, 1982. *Justus Liebigs Ann. Chem.*, 845-852.

I. Monoclinic,  $P2_1/b$ ,  $a = 9.511$ ,  $b = 14.109$ ,  $c = 15.474$  Å,  $\gamma = 110.18^\circ$ ,  $D_m = 1.71$ ,  $Z = 4$ . Cu radiation,  $R = 0.070$  for 2583 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 8.774$ ,  $b = 9.523$ ,  $c = 7.151$  Å,  $\alpha = 96.81$ ,  $\beta = 100.83$ ,  $\gamma = 61.29^\circ$ ,  $D_m = 1.67$ ,  $Z = 1$ . Cu radiation,  $R = 0.070$  for 1872 reflexions.

III. Monoclinic,  $P2_1/b$ ,  $a = 10.097$ ,  $b = 14.330$ ,  $c = 15.530$  Å,  $\gamma = 103.00^\circ$ ,  $D_m = 1.59$ ,  $Z = 4$ . Cu radiation,  $R = 0.064$  for 3369 reflexions.

Molecule II (Fig. 1) is centrosymmetric while in its isomer (III) the fused ring system has approximate  $C_2$ -symmetry. In both molecules the five-membered rings adopt an envelope conformation. [No discussion or figure is given for (I).]

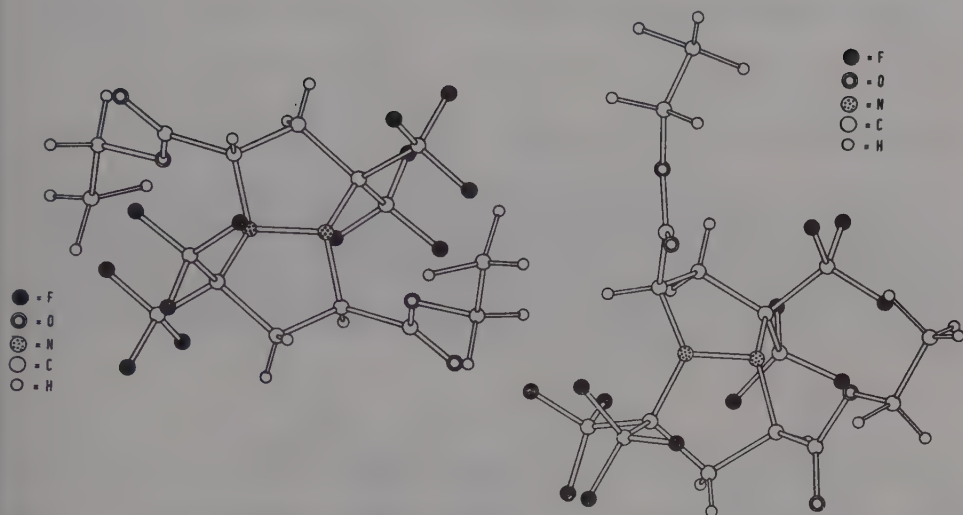
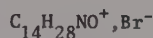


Fig. 1. Perspective views of the trans (II) and cis (III) forms of  $C_{16}H_{16}F_{12}N_2O_4$ .

(±)-2-DEPENTYLPYRHYDROHISTRIONICOTOXIN HYDROBROMIDE  
 $C_{14}H_{28}BrNO$



K. TAKAHASHI, A.E. JACOBSON, C.-P. MAK, B. WITKOP, A. BROSSI, E.X. ALBUQUERQUE, J.E. WARNICK, M.A. MALEQUE, A. BAVOSO and J.V. SILVERTON, 1982. *J. Med. Chem.*, **25**, 919-925.

Triclinic,  $P\bar{1}$ ,  $a = 7.902$ ,  $b = 8.271$ ,  $c = 13.299$  Å,  $\alpha = 103.03$ ,  $\beta = 94.12$ ,  $\gamma = 113.81^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.037$  for 2537 reflexions.

In the cation the hydroxy oxygen atom is axial (Fig. 1) and torsion angles of the piperidine ring correspond to an almost ideal chair conformation. Packing in the crystal is strongly influenced by hydrogen bonding and electrostatic attractions. There is an intramolecular hydrogen bond  $N-H \cdots O$  (hydroxy) with an  $N \cdots O$  length of 2.774 Å, an  $H \cdots O$  distance of 2.165 Å, and an  $N-H \cdots O$  angle of  $132^\circ$ . This H atom forms a bifurcated bond since it also interacts with a second O (hydroxy) atom in a molecule related by a centre of symmetry on the  $ab$  plane:  $N \cdots O$  2.975 Å,  $H \cdots O$  2.313 Å,  $N-H \cdots O$   $138^\circ$ . The OH group is also hydrogen bonded to the  $Br^-$  anion ( $O \cdots Br$  3.258,  $H \cdots Br$  2.596 Å,  $O-H \cdots Br$   $161^\circ$ ). The second amino H atom forms a hydrogen bond to a neighbouring  $Br^-$ ,  $N \cdots Br$  3.320,  $H \cdots Br$  2.491 Å,  $N-H \cdots Br$   $172^\circ$ .

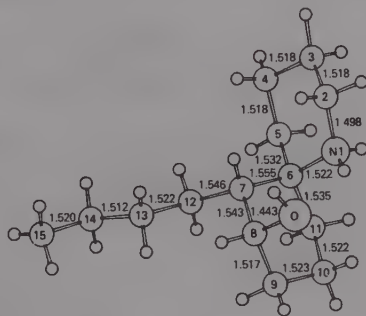


Fig. 1. Molecular structure of  $C_{14}H_{28}NO^+$  showing bond lengths in Å.



## 2-(2-INDOLYL)-3-AMINO-4(3H)-QUINAZOLINONE

J. BERGMAN and N. EKLUND, 1982. Chem. Scr., 19, 193-204.

Monoclinic,  $A2/m$ ,  $a = 13.167$ ,  $b = 8.790$ ,  $c = 22.42 \text{ \AA}$ ,  $\beta = 90.21^\circ$ ,  $D_m = 1.4$ ,  $Z = 8$ .  
Mo radiation,  $R = 0.046$  for 1113 reflexions.

The whole molecule is approximately coplanar except for the H atoms on N(4) (Fig. 1). The C(6)-C(14) bond between the two ring systems in the molecule is too long ( $1.457 \text{ \AA}$ ) to be a true  $\pi$ -bond but also too short to be a true  $\sigma$ -bond. The C(1)-C(7) bond length of  $1.218 \text{ \AA}$  indicates double bond character, and the N(3)-N(4) bond length of  $1.423 \text{ \AA}$  is too long for any significant conjugation of the lone pair orbital on N(4) with the quinazolinone ring system. Steric repulsion between N(4) and H(15) (N...H  $2.38 \text{ \AA}$ ) results in angles C(6)-C(14)-C(15) and C(6)-C(14)-N(5) assuming values of  $136.2$  and  $115.4^\circ$  respectively.

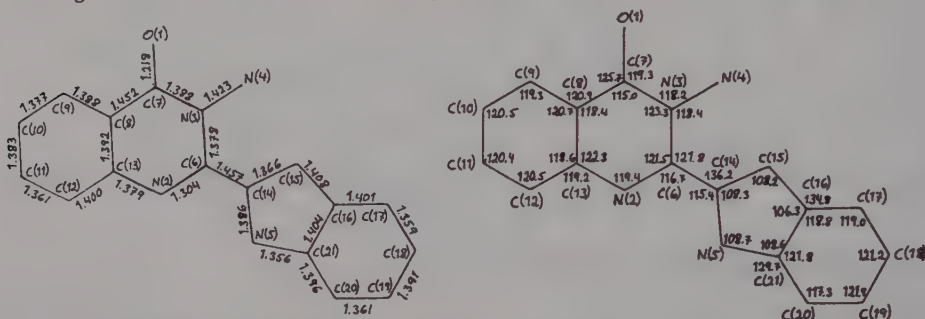


Fig. 1. Bond lengths ( $\text{\AA}$ ), and bond angles ( $^\circ$ ) in  $C_{16}H_{12}N_4O$ .

## 7-CHLORO-5-PHENYL-3(S)-METHYL-2H-1,4-BENZODIAZEPINE-2-ONE

M. SIKIRICA and I. VICKOVIC, 1982. Cryst. Struct. Comm., 11, 1293-1298.

Monoclinic,  $P2_1$ ,  $a = 20.860$ ,  $b = 7.523$ ,  $c = 14.856 \text{ \AA}$ ,  $\beta = 109.68^\circ$ ,  $Z = 6$ . Mo radiation,  $R = 0.072$  for 2103 reflexions.

The three independent molecules in the asymmetric unit have similar geometries. The seven-membered ring (Fig. 1) is in boat conformation, with methyl at C3 quasi-equatorial. Molecules are linked by N-H...O hydrogen bonds ( $2.88$  and  $2.82(2) \text{ \AA}$ ).

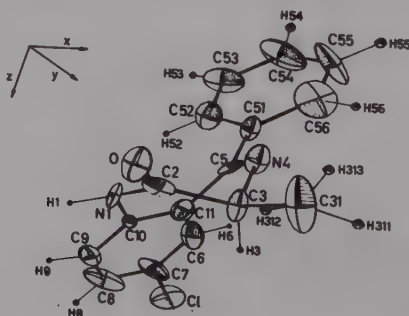


Fig. 1. A  $C_{16}H_{13}ClN_2O$  molecule.

7-CHLORO-2-METHYLAMINO-5-PHENYL-3H-1,4-BENZODIAZEPINE 4-OXIDE (CHLORDIAZEPOXIDE)  
 $C_{16}H_{14}ClN_3O$

V. BERTOLASI, M. SACERDOTI, G. GILLI and P.A. BOREA, 1982. Acta Cryst., **B38**, 1768-1772.

Triclinic,  $P\bar{1}$ ,  $a = 15.786$ ,  $b = 13.155$ ,  $c = 15.496$  Å,  $\alpha = 104.56$ ,  $\beta = 102.43$ ,  $\gamma = 79.83^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.055$  for 5829 reflexions.

The asymmetric unit consists of four molecules, all with the same absolute configuration, linked into dimers by two pairs of hydrogen bonds ( $N...O$  2.788(7) - 2.910(7) Å). The rotation of the C(11)-C(16) phenyl group (Fig. 1) around C(9)-C(11) is in the range 40.3-45 Å for all four molecules. The diazepine ring adopts a boat conformation with different twist components in each molecule. Some bond lengths are: N(1)-C(7) 1.299(2), N(2)-C(9) 1.314(2), N-O 1.310(3), C(1)-C1 1.745(5) Å.

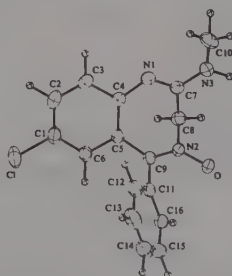


Fig. 1. One of the four independent chlordiazepoxide molecules.

3-PHENYL-5-OXO-7,7-DIMETHYL-1(H),4(H)-5,6,7,8-TETRAHYDROBENZO[c]PYRIDAZINE  
 $C_{16}H_{18}N_2O$

P.V. PADMANABHAN, S.R. RAMADAS, B. VARGHESE and S. SRINIVASAN, 1982. Cryst. Struct. Comm., **11**, 1277-1282.

Orthorhombic,  $Pbca$ ,  $a = 9.158$ ,  $b = 11.424$ ,  $c = 25.783$  Å,  $D_m = 1.250$ ,  $Z = 8$ . Cu radiation,  $R = 0.059$  for 652 reflexions.

The analysis establishes the structure of this reaction product to be as shown in Fig. 1.

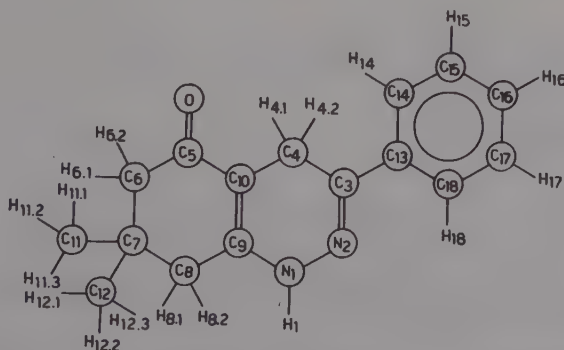


Fig. 1. The  $C_{16}H_{18}N_2O$  molecule.

7-ISOBUTYL-5-METHYL-2-PHENYLIMIDAZO[5,1-f][1,2,4]TRIAZIN-4(3H)-ONE  
 $C_{16}H_{18}N_4O$

P. MURRAY-RUST, J. MURRAY-RUST and A.W. OXFORD, 1982. Acta Cryst., B38, 2692-2693.

Monoclinic,  $P2_1/n$ ,  $a = 15.03$ ,  $b = 17.40$ ,  $c = 5.74 \text{ \AA}$ ,  $\beta = 93.81^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.056$  for 1131 reflexions.

In the molecule (Fig. 1) the side chain is nearly perpendicular to the heterocyclic part and the isopropyl group has a trans-gauche arrangement. The structure consists of hydrogen-bonded molecules about a centre of symmetry  $N(3) \dots O(1')$   $2.86(1) \text{ \AA}$ .

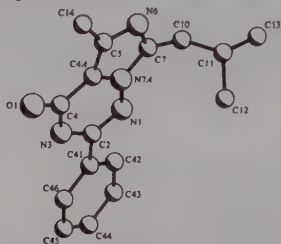


Fig. 1.  $C_{16}H_{18}N_4O$ : perspective view of the molecule.

(3R,6S)-3-BENZYL-4-METHYL-1,4-DIAZA-BICYCLO[4.4.0]DECAN-2,5-DIONE  
 $C_{16}H_{20}N_2O_2$

M. VAN POUCKE and A.T.H. LENSTRA, 1982. Bull. Soc. Chim. Belg., 91, 213-218.

Orthorhombic,  $P2_12_12_1$ ,  $a = 10.121$ ,  $b = 10.941$ ,  $c = 13.390 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.086$  for 927 reflexions.

The piperidine ring is in chair form (Fig. 1) and the deketopiperazine ring is in a shallow boat form. The benzyl group is axially oriented and is folded back over the deketopiperazine ring.

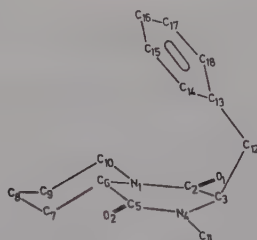


Fig. 1. The  $C_{16}H_{20}N_2O_2$  molecule.

8-CHLORO-1-METHYL-6-PHENYL-1,2,3,4-TETRAHYDRO-1,5-BENZODIAZOCIN-2-ONE  
 $C_{17}H_{15}ClN_2O$

A.A. DVORKIN, Yu.A. SIMONOV, T.I. MALINOWSKY, S.S. ANDRONATI, A.V. BOGATSKY and V.V. DANILIN, 1982. Acta Cryst., B38, 638-640.

Orthorhombic,  $P2_12_12_1$ ,  $a = 13.374$ ,  $b = 15.951$ ,  $c = 7.056 \text{ \AA}$ ,  $Z = 4$ . Mo radiation,  $R = 0.079$  for 1089 reflexions.

The eight-membered ring has a boat conformation. The two phenyl rings are inclined at  $85.2^\circ$  to each other. Angles at C(8) and C(9) are about  $116^\circ$ . There are no short intermolecular contacts.

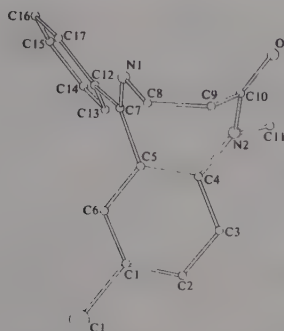


Fig. 1. The molecule of 8-chloro-1-methyl-6-phenyl-1,2,3,4-tetrahydro-1,5-benzodiazocin-2-one.

1,2-((CARBONYLAMINO)CARBONYL)-18-(3,4-DICHLOROPHENYL)-6 $\alpha$ -HYDROXY-N-METHYL-4-CYCLO-  
HEXENYL ACETATE

$C_{17}H_{15}Cl_2NO_4$

T.C. MCKENZIE, W.J. FANSHAW, J.W. EPSTEIN and J.B. COLLINS, 1982. J. Org. Chem., 47, 352-354.

Monoclinic,  $P2_1/n$ ,  $a = 11.694$ ,  $b = 8.408$ ,  $c = 17.566$  Å,  $\beta = 99.58^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.041$  for 2574 reflexions.

X-ray analysis was used to confirm the molecular structure of the Diels-Alder acetate adduct (Fig. 1), as determined by molecular mechanics methods. The cyclohexene ring has a distorted boat conformation, with the acetate group trans to the phenyl ring and in a prow (axial) position. The only difference between the X-ray structure and that found from NMR data and energy calculations was in the twisting of the methylene carbon, C12, away from the acetate group, causing a flattening of the cyclohexene ring.

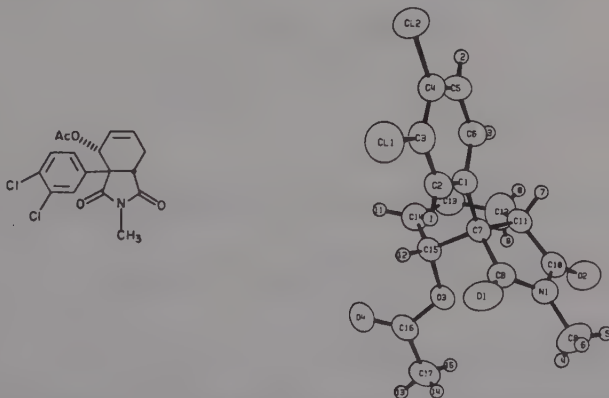


Fig. 1. Formula and view of a molecule of the acetate,  $C_{17}H_{15}Cl_2NO_4$ .

1,2,5-TRIMETHYL-3-PHENYL-2H-ISOINDOLE-4,7-DIONE

$C_{17}H_{15}NO_2$

P. SINGH, L. SCOTT and J.A. MYERS, 1982. Cryst. Struct. Comm., 11, 1091-1098.

The analysis establishes the structure as shown in Fig. 1. The plane of the phenyl ring is inclined at  $57.4^\circ$  to the heterocyclic ring.

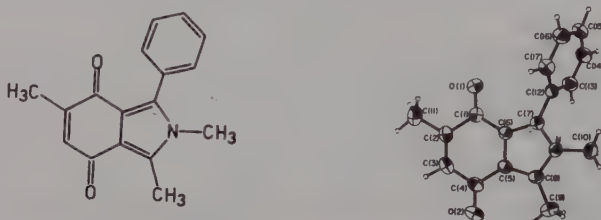


Fig. 1. The  $C_{17}H_{15}NO_2$  molecule.

F. FLORENCIO, P. SMITH-VERDIER and S. GARCÍA-BLANCO, 1982. *Acta Cryst.*, **B38**, 2301-2304.

Orthorhombic,  $Pbca$ ,  $a = 24.520$ ,  $b = 11.795$ ,  $c = 10.577$  Å,  $Z = 8$ . Mo radiation,  $R = 0.078$  for 1528 reflexions.

In the molecule (Fig. 1) the cyclohexane ring of the bicyclic system adopts a highly distorted boat conformation, the five-membered ring adopts a 6,1-half-chair conformation, and the cyclohexane ring substituted at N(3') has a highly symmetrical chair conformation. The succinimide ring is planar and the symmetry of the whole molecule approximates to  $m$  with a mirror plane through the succinimide ring. Intermolecular contacts correspond to normal van der Waals interactions.

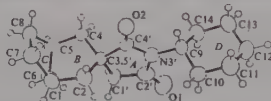


Fig. 1.  $C_{17}H_{25}NO_2$ : a perspective view of the molecule.

V. BERTOLASI, V. FERRETTI, G. GILLI and P.A. BOREA, 1982. *Cryst. Struct. Comm.*, **11**, 1481-1486.

Monoclinic,  $P2_1/c$ ,  $a = 9.467$ ,  $b = 14.225$ ,  $c = 13.421$  Å,  $\beta = 100.62^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.069$  for 1774 reflexions.

The diazepin ring (Fig. 1) has a boat conformation and atom N(1) is trigonal planar. The N(2)-C(9) bond (1.269(7) Å) corresponds to a double bond. The ethoxy chain is almost zig-zag planar, and the chloroethyl chain is folded reflecting an electrostatic intramolecular interaction between N(1) and C1(2) (N...C1 3.123(5) Å).

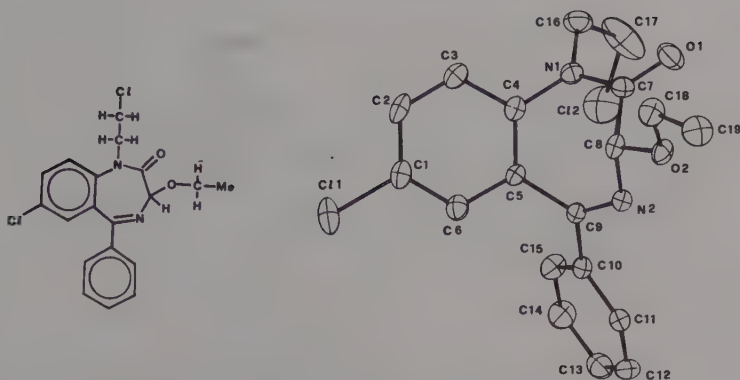


Fig. 1. A view of  $C_{19}H_{18}Cl_2N_2O_2$ .

2-{2-[4-(DIMETHYLAMINO)PHENYL]ETHENYL}QUINOLINE

$C_{19}H_{18}N_2$

O.I. LEVINA, V.G. RAU, K.A. POTEKHIN, E.N. KURKUTOVA, L.G. PAKHOMOV and Yu.T. STRUCHKOV, 1982. Cryst. Struct. Comm., 11, 1899-1902.

Orthorhombic,  $Pna2_1$ ,  $a = 23.351$ ,  $b = 11.227$ ,  $c = 5.811$  Å,  $Z = 4$ . Mo radiation,  $R = 0.048$  for 789 reflexions.

The molecule (Fig. 1) is almost planar; the benzene ring and dimethylamino group form a dihedral angle of  $5.7^\circ$ . Bond lengths are normal.

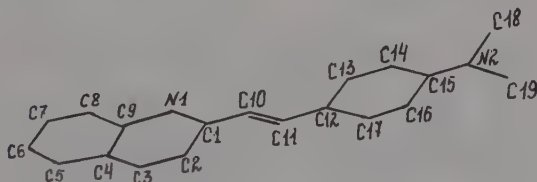


Fig. 1. The  $C_{19}H_{18}N_2$  molecule.

cis-(S)-(+)-6'-BROMO-N-FORMYLNORRETICULINE

$C_{19}H_{20}BrNO_5$

P. BUCHS, K.C. RICE, A. BROSSI, J.V. SILVERTON and R. POTENZONE, JR., 1982. J. Org. Chem., 47, 4134-4137.

Monoclinic,  $P2_1$ ,  $a = 6.616$ ,  $b = 10.985$ ,  $c = 13.084$  Å,  $\beta = 95.61^\circ$ ,  $D_m = 1.47$ ,  $Z = 2$ . Cu radiation,  $R = 0.043$  for 3151 reflexions.

The analysis establishes the S-configuration for the molecule (Fig. 1). Bond lengths and angles are normal and packing is controlled by hydrogen bonding.



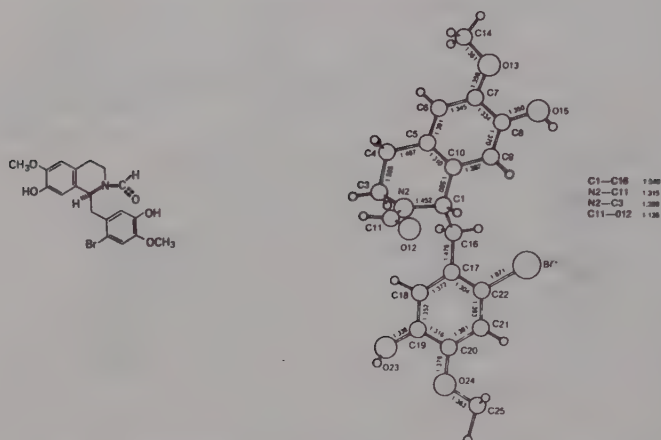


Fig. 1. A view of  $C_{19}H_{20}BrNO_5$ .

(1R,5R,6S,7R)-1-BENZOYLOXY-7-ETHOXYCARBONYL-5-METHYL-8-AZABICYCLO[4.3.0]NONANE-4,9-DIONE  
 $C_{19}H_{21}NO_6$

H.M. DOESBURG, J.H. NOORDIK, J.M. LEMMENS and L. THIJS, 1982. Cryst. Struct. Comm., 11, 557-560.

Monoclinic,  $P2_1/c$ ,  $a = 11.034$ ,  $b = 11.697$ ,  $c = 14.823$  Å,  $\beta = 110.21^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.039$  for 968 reflexions.

Bond lengths and angles in this molecule (Fig. 1) have acceptable values. There are only van der Waals interactions between molecules.



Fig. 1. A stereoview of  $C_{19}H_{21}NO_6$ .

(1R,5R,6S,7S)-1-BENZOYLOXY-7-ETHOXYCARBONYL-5-METHYL-8-AZABICYCLO[4.3.0]NONANE-4,9-DIONE  
 $C_{19}H_{21}NO_6$

H.M. DOESBURG, J.H. NOORDIK, J.M. LEMMENS and L. THIJS, 1982. Cryst. Struct. Comm., 11, 551-555.

Monoclinic,  $P2_1/c$ ,  $a = 10.825$ ,  $b = 10.975$ ,  $c = 15.426$  Å,  $\beta = 103.49^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.062$  for 647 reflexions.

Geometry details are normal except for dimensions about C13 (Fig. 1) which has large thermal motion. Molecules are linked by N-H...O hydrogen bonds to form dimers (N...O 3.12(1) Å).

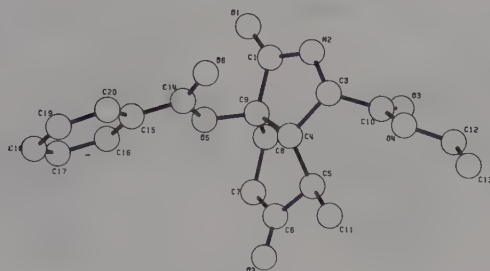


Fig. 1. A view of  $C_{19}H_{21}NO_6$ .

(6R)-PENTAACETYL-5,6,7,8-TETRAHYDRO-L-BIOPTERIN ETHYL ACETATE SOLVATE  
 $C_{19}H_{25}N_5O_8 \cdot C_4H_8O_2$

R. PREWO, J.H. BIERI, S.N. GANGULY and M. VISCONTI, 1982. *Helv. Chim. Acta*, **65**, 1094-1099.

Orthorhombic,  $P2_12_12_1$ ,  $a = 8.053$ ,  $b = 14.955$ ,  $c = 21.502$  Å,  $Z = 4$ . Mo radiation,  $R = 0.093$  for 4240 reflexions (at  $-140^\circ\text{C}$ ).

The analysis establishes the structure and dimensions as shown in Fig. 1.

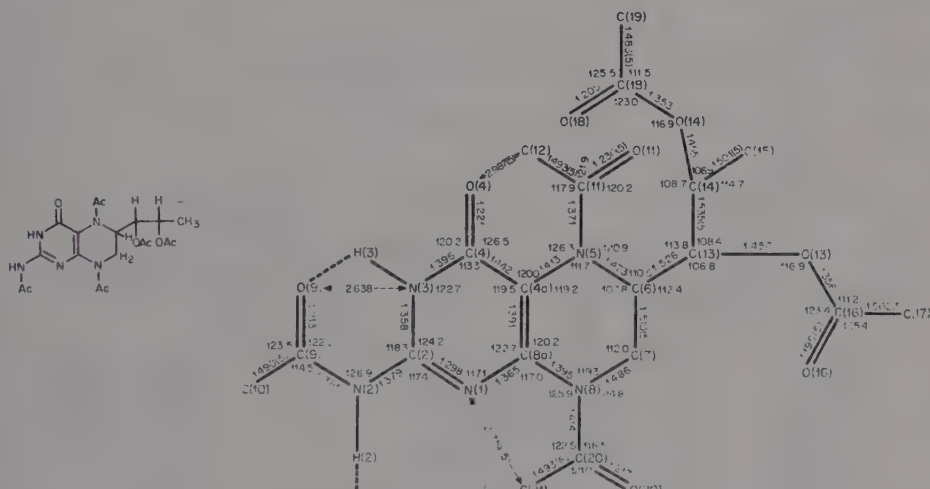


Fig. 1. The  $C_{19}H_{25}N_5O_8$  molecule and dimension.

2-(DIPHENYLMETHOXYCARBONYL)-3-METHYL-1-AZABICYCLO[3.2.0]HEPTANE-4,7-DIONE  
 $C_{21}H_{19}NO_4$

R.L. ROSATI, L.V. KAPILI, P. MORRISSEY, J. BORDNER and E. SUBRAMANIAN, 1982. *J. Am. Chem. Soc.*, **104**, 4262-4264.

Monoclinic,  $P2_1$ ,  $a = 8.275$ ,  $b = 5.796$ ,  $c = 19.409$  Å,  $\beta = 102.36^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.065$ .

The molecule is shown in Fig. 1. The assignment of absolute configuration rests on the assumption that the configuration at C(5) is the same as that in the synthetic

precursor. No unusual structural features were noted.

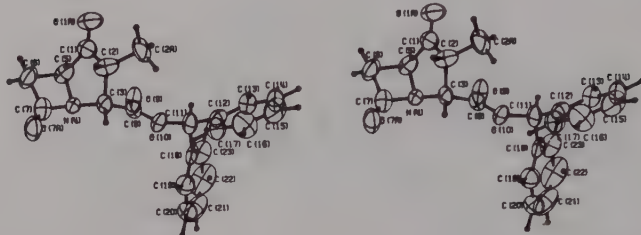


Fig. 1. Stereoview of  $C_{21}H_{19}NO_4$ .

(3aR,7R,7aR)-N-BENZYL-2-OXO-7-PHENYL-2,3,3a,6,7,7a-HEXAHYDROINDOLE  
 $C_{21}H_{21}NO$  (I)

(3aR,7R,7aS)-N-BENZYL-2-OXO-7-PHENYL-2,3,3a,6,7,7a-HEXAHYDROINDOLE  
 $C_{21}H_{21}NO$  (II)

S.F. MARTIN, C.-Y. TU, M. KIMURA and S.H. SIMONSEN, 1982, *J. Org. Chem.*, **47**, 3634-3643.

I. Monoclinic,  $P2_1/c$ ,  $a = 14.223$ ,  $b = 8.621$ ,  $c = 14.853$  Å,  $\beta = 116.96^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.038$  for 2650 reflexions (at 180 K).

II. Triclinic,  $P\bar{1}$ ,  $a = 9.415$ ,  $b = 11.815$ ,  $c = 8.858$  Å,  $\alpha = 96.85^\circ$ ,  $\beta = 107.55^\circ$ ,  $\gamma = 114.66^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.039$  for 2695 reflexions (at 180 K).

The analyses unequivocally established the stereochemistry of these two isomers as shown in Fig. 1.

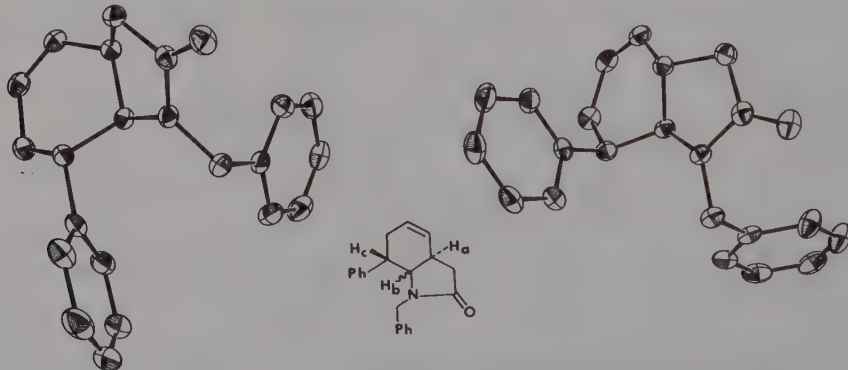


Fig. 1. Views of the  $C_{21}H_{21}NO$  molecules (I) (left), (II) (right) (I  $H_b = \alpha$ , II  $H_b = \beta$ ).

(3R,4R,5S)-4-[[2-(METHOXYCARBONYL)ETHYL]SULPHINYL]-1-[1-(METHOXYCARBONYL)-2-METHYL-PROP-1-ENYL]-3-PHTHALIMIDO-2-AZETIDINONE  
 $C_{21}H_{22}N_2O_8S$

M.D. BACHI, A. GROSS and F. FROLOW, 1982, *J. Org. Chem.*, **47**, 765-767.

Orthorhombic,  $P2_12_12_1$ ,  $a = 10.745$ ,  $b = 13.874$ ,  $c = 14.934$  Å,  $D_m = 1.375$ ,  $Z = 4$ . Mo radiation,  $R = 0.09$  for 2500 reflexions.

The analysis establishes that the molecule has the configuration shown in Fig. 1 for atoms C-3 and C-4.

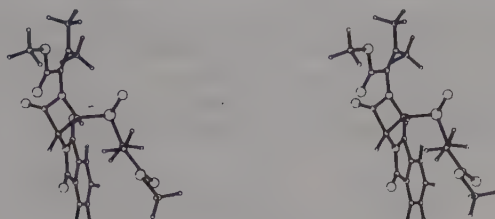
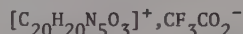
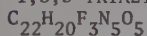


Fig. 1. A stereoview of  $C_{21}H_{22}N_2O_8S$ .

4-OXO-3H-2-[N-(p-METHOXYPHENYL) IMINIUM] -6-METHYL-8-(p-METHOXYPHENYL) IMIDAZO[1,2-a]-1,3,5-TRIAZINE TRIFLUOROACETATE



M.R. UDUPA and B. KREBS, 1982. Z. Krist., 158, 149-157.

Monoclinic,  $P2_1/c$ ,  $a = 10.404$ ,  $b = 12.997$ ,  $c = 16.511$  Å,  $\beta = 92.45^\circ$ ,  $D_m = 1.44$ ,  $Z = 4$ . Mo radiation,  $R = 0.060$  for 2387 reflexions.

The nitrogen atom of the methoxyphenylimine group (Fig. 1) is protonated to give the univalent cation. The methyl imidazotriazinone ring system and the methoxyphenyl and the methoxyphenyliminium groups are all planar but are twisted with respect to each other. Strong hydrogen bonding is observed between the carboxylate oxygen atoms of the  $CF_3COO^-$  ion and the two NH groups of the cation, one of the bonds being extremely short ( $O \cdots N$  2.666 Å).

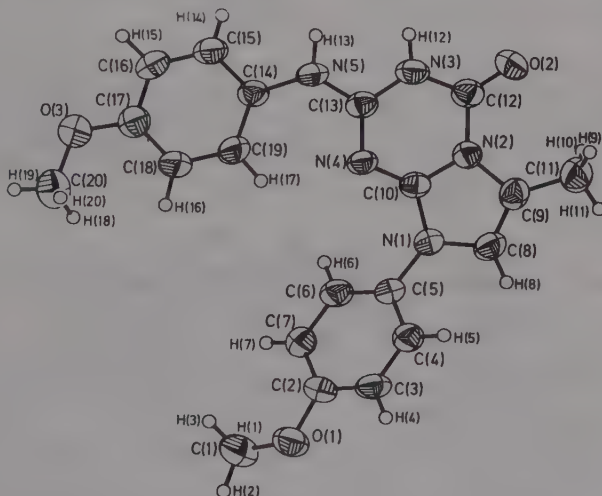
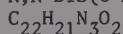


Fig. 1. The  $[C_{20}H_{20}N_5O_3]^+$  cation.

N,N-BIS(8-HYDROXY-2-QUINOLYL)-n-BUTYLAMINE



I.N. POLYAKOVA, I.A. TESLYA, Z.A. STARIKOVA, B.V. PARUSNIKOV and I.A. KRASAVIN, 1982. Kristallografiya, 27, 496-500 [Sov. Phys. Crystallogr., 27, 301-303].



Monoclinic,  $P2_1/c$ ,  $a = 12.177$ ,  $b = 14.553$ ,  $c = 13.014$  Å,  $\beta = 111.38^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.079$  for 1291 reflexions.

The structure of this unusual 1:2 carbenemethacrylonitrile adduct was found to incorporate a 1'-azaspiro[5,4] system. No unusual bond lengths or angles were noted.

7-CHLORO-5-PHENYL-1-[(S)-1-PHENYLETHYL]-1,3-DIHYDRO-2H-1,4-BENZODIAZEPIN-2-ONE  
 $C_{23}H_{19}ClN_2O$  (I)

7-CHLORO-5-PHENYL-3-METHYL-1-[(S)-1-PHENYLETHYL]-1,3-DIHYDRO-2H-1,4-BENZODIAZEPIN-2-ONE  
 $C_{24}H_{21}ClN_2O$  (II)

Ž. RUŽIĆ-TOROŠ, B. KOJIĆ-PRODIĆ, N. BRESCIANI-PAHOR, G. NARDIN and L. RANDACCIO, 1982. *Acta Cryst.*, B38, 2977-2981.

I. Orthorhombic,  $P2_12_12_1$ ,  $a = 17.500$ ,  $b = 12.036$ ,  $c = 9.212$  Å,  $Z = 4$ . Mo radiation,  $R = 0.047$  for 1778 reflexions.

II. Orthorhombic,  $P2_12_12_1$ ,  $a = 15.912$ ,  $b = 14.403$ ,  $c = 8.835$  Å,  $Z = 4$ . Mo radiation,  $R = 0.029$  for 1482 reflexions.

Bond lengths and angles in I and II (Fig. 1) are generally as expected. The benzodiazepine ring exhibits a boat form with a P absolute configuration in both compounds. The absolute configuration at C(1) is S (in I and II) whereas at the induced chiral centre C(3) (in II) it is R.

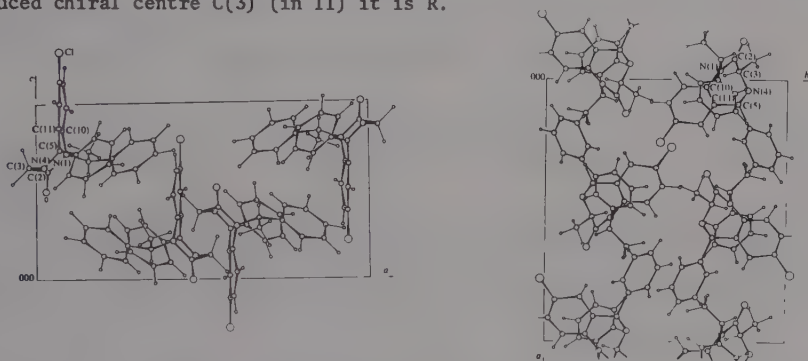


Fig. 1.  $C_{23}H_{19}ClN_2O$  (I) and  $C_{24}H_{21}ClN_2O$  (II): views of the crystal structures of I (along b) and II (along c).

cis-N-3-HYDROXYPROPYL-5'-METHOXYLAUDANOSINIUM IODIDE MONOHYDRATE  
 $C_{25}H_{36}INO_6 \cdot H_2O$  (I)

trans-N-3-HYDROXYPROPYL-5'-METHOXYLAUDANOSINIUM PERCHLORATE  
 $C_{25}H_{36}ClNO_{10}$  (II)

H.A. EL-SAYAD, R.A. SWARINGEN, D.A. YEOWELL, R.C. CROUCH, S. HURLBERT, R.W. MILLER and A.T. McPHAIL, 1982. *J. Chem. Soc. Perkin I*, 2067-2077.

I. Monoclinic,  $P2_1/c$ ,  $a = 11.475$ ,  $b = 17.749$ ,  $c = 14.278$  Å,  $\beta = 113.51^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.077$  for 2987 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 8.123$ ,  $b = 20.380$ ,  $c = 16.710$  Å,  $\beta = 102.14^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.082$  for 2215 reflexions.

The relative stereochemistry of the two isomers was determined by the X-ray



structure analysis. The nitrogen containing ring in I is close to an envelope conformation, leading to a decrease in non-bonded interactions between the benzyl and isopropyl substituents. This ring in II is closer to a half chair conformation.

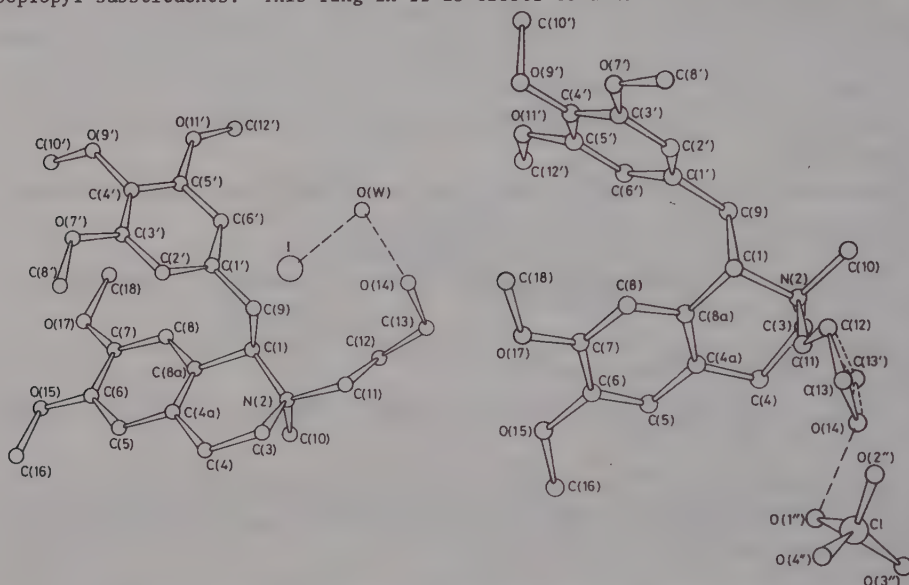


Fig. 1. The molecular structures of  $C_{25}H_{36}INO_6 \cdot H_2O$  (I) (left) and  $C_{25}H_{36}ClNO_{10}$  (II) (right).

1'-(4-BROMOBENZOYL)-2',3'-DIHYDRO-2,3,3',3'-TETRAMETHYL-1,2'-BI-1H-INDOLE  
 $C_{27}H_{25}BrN_2O$

K. HARANO, M. YASUDA and T. TAGUCHI, 1982. *Cryst. Struct. Comm.*, **11**, 1223-1226.

Orthorhombic,  $Pbca$ ,  $a = 17.273$ ,  $b = 15.608$ ,  $c = 17.038$  Å,  $D_m = 1.386$ ,  $Z = 8$ . Mo radiation,  $R = 0.078$  for 1324 reflexions.

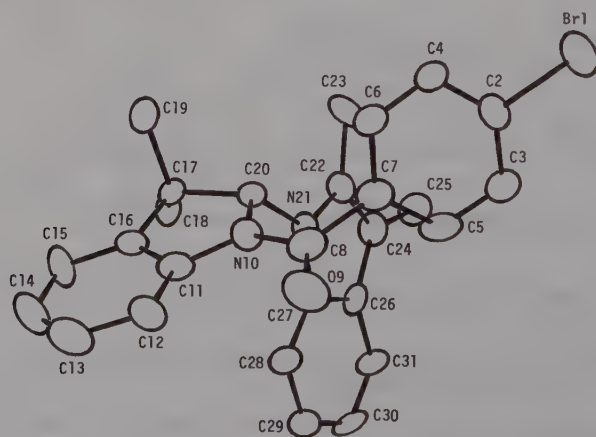
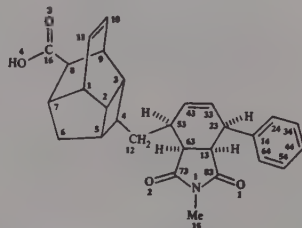


Fig. 1. A view of  $C_{27}H_{25}BrN_2O$ .

In this molecule (Fig. 1) the indole ring is almost planar with a maximal displacement of 0.04 Å. The carbon atoms C(17) and C(20) lie 0.12 Å below and 0.10 Å above the best plane through atoms C(11), C(12), C(13), C(14), C(15), and C(16) respectively. Atoms C(8), O(9), N(10), C(11) and C(12) are roughly coplanar, suggesting some conjugation along the -CO-N-Ar system. The dihedral angle between the plane of the indole ring and the plane of the indoline ring is near 85°.

4-((2''-METHYL-1'',3''-DIOXO-7''-PHENYL-1'',3'',3a'',4'',7'',7a''-HEXAHYDROISOINDOL-4''-YL)METHYL)TETRACYCLO[5.4.0.0<sup>2</sup>,5.0<sup>3</sup>,9]UNDEC-10-ENE-8-CARBOXYLIC ACID

C<sub>28</sub>H<sub>29</sub>NO<sub>4</sub>



W.M. BANDARANAYAKE, J.E. BANFIELD, D. St.C. BLACK, G.D. FALLON and B.M. GATEHOUSE, 1982. *Aust. J. Chem.*, **35**, 567-579.

Triclinic,  $P\bar{1}$ ,  $a = 14.214$ ,  $b = 9.698$ ,  $c = 10.744$  Å,  $\alpha = 97.93$ ,  $\beta = 125.21$ ,  $\gamma = 97.16^\circ$ ,  $D_m = 1.29$ ,  $Z = 2$ . Cu radiation,  $R = 0.052$  for 1974 reflexions.

The structure was confirmed as that shown above with normal values for bond lengths and angles.

n-BUTYL-2-(2-OXO-3-[N-(2-OXO-2,3-DIHYDRO-5-BENZIMIDAZOLYL)-CARBAMOYL]NAPHTHYLIDEN-HYDRAZINO)BENZOATE

C<sub>29</sub>H<sub>25</sub>N<sub>5</sub>O<sub>5</sub>

E.F. PAULUS, 1982. *Z. Krist.*, **160**, 235-243.

Triclinic,  $P\bar{1}$ ,  $a = 13.038$ ,  $b = 9.996$ ,  $c = 11.853$  Å,  $\alpha = 108.91$ ,  $\beta = 117.30$ ,  $\gamma = 98.53^\circ$ ,  $D_m = 1.39$ ,  $Z = 2$ . Cu radiation,  $R = 0.073$  for 3353 reflexions.

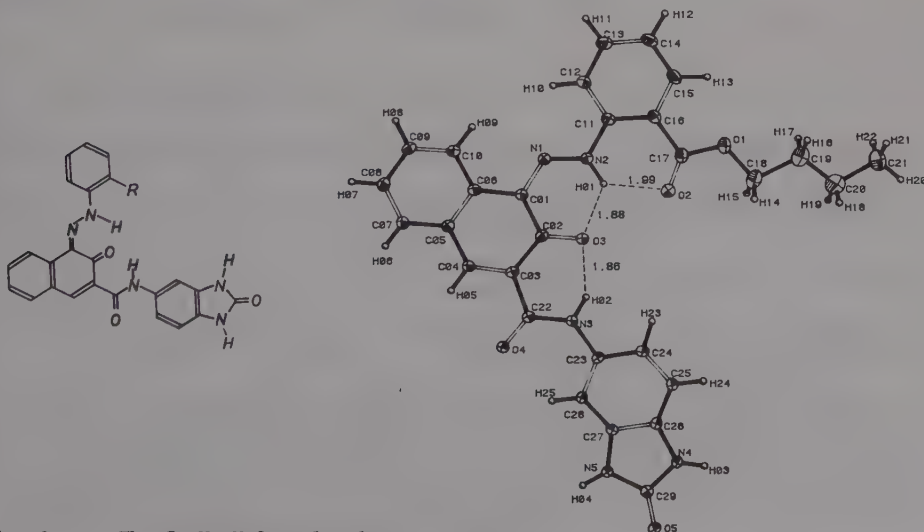


Fig. 1. The C<sub>29</sub>H<sub>25</sub>N<sub>5</sub>O<sub>5</sub> molecule.

The analysis establishes that the molecule is a hydrazone with anticipated bond lengths and angles. There are three intramolecular hydrogen bonds (Fig. 1) and two intermolecular ones within layers parallel to (023).

ETHYL trans-7-BENZYLOXY-4-HYDROXY-6-METHOXY-1-(3,4,5-TRIMETHOXYPHENETHYL)-1,2,3,4-TETRAHYDRO-2-ISOQUINOLINECARBOXYLATE

$C_{31}H_{37}NO_8$

S. BELLARD, R. ELLIOTT and E. McDONALD, 1982. Acta Cryst., B38, 669-671.

Triclinic,  $P\bar{1}$ ,  $a = 12.700$ ,  $b = 12.045$ ,  $c = 10.699$  Å,  $\alpha = 107.47^\circ$ ,  $\beta = 112.79^\circ$ ,  $\gamma = 83.25^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.048$  for 4027 reflexions.

The molecule (Fig. 1) has the 1 and 4 substituents in pseudo-axial positions. The 3' and 5' methoxy groups are nearly coplanar with the benzene ring while the 4' group is at  $99.4^\circ$  to the ring plane. The C-O-CH<sub>3</sub> angles at 3', 4' and 5' are  $119.1^\circ$ ,  $112.1^\circ$  and  $117.2^\circ$  respectively. Molecules are linked by O(16)-H...O(11) hydrogen bonds.

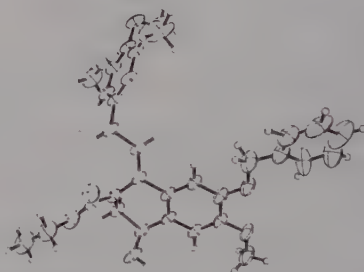
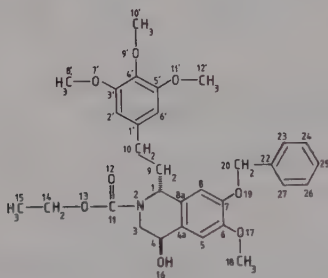


Fig. 1. The  $C_{31}H_{37}NO_8$  molecule.

1,3-BIS(1,3,3-TRIETHYLINDOLINE-2-YL)TRIMETHINIUM PERCHLORATE

$C_{31}H_{41}ClN_2O_4$  (I)

1,3-BIS(1,3,3-TRIETHYLINDOLINE-2-YL)-2-AZATRIMETHINIUM TETRAFLUOROBORATE

$C_{30}H_{40}BF_4N_3$  (II)

1,3-BIS(1,3,3-TRIETHYLINDOLINE-2-YL)-2-PHOSPHATRIMETHINIUM TETRAFLUOROBORATE

$C_{30}H_{40}BF_4N_2P$  (III)

R. ALLMANN, A. WASKOWSKA and S. OLEJNIK, 1982. Cryst. Struct. Comm., 11, 1077-1082.

I. Monoclinic,  $P2_1/c$ ,  $a = 14.714$ ,  $b = 17.222$ ,  $c = 13.313$  Å,  $\beta = 117.23^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.098$  for 3077 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 14.265$ ,  $b = 17.123$ ,  $c = 13.480$  Å,  $\beta = 116.70^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.072$  for 3577 reflexions.

III. Monoclinic,  $P2_1/c$ ,  $a = 15.080$ ,  $b = 17.548$ ,  $c = 13.171$  Å,  $\beta = 119.41^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.108$  for 2284 reflexions.

All three cyanine cations (Fig. 1) are in the all-trans form and all are very similar. They differ only in the C-Z distances and C-Z-C angles: C-C 1.38, C-N 1.34 and C-P 1.71 Å; C-C-C 123.1°, C-N-C 116.1° and C-P-C 101.8°. The dihedral angles between the two indoline moieties are  $13.8^\circ$ ,  $13.3^\circ$  and  $15.1^\circ$  respectively. All three cyanine cations possess a pseudo-twofold axis. All ethyl groups exhibit a staggered conformation and are about perpendicular to the indoline planes. The corrected mean C1-O and B-F bond lengths amount to 1.44, 1.39 and 1.42 Å.

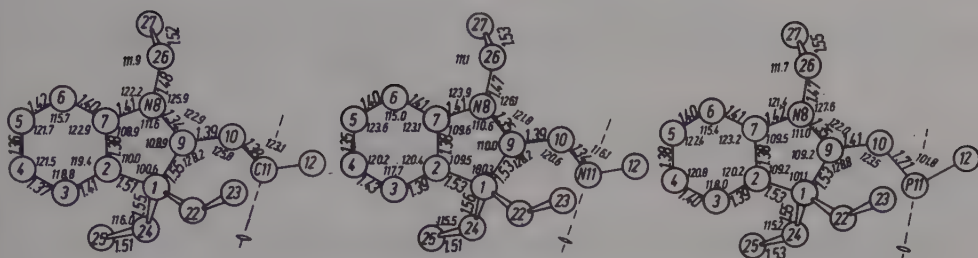


Fig. 1. Averaged bond angles and bond lengths (lengths corrected for thermal vibration) for the three cyanine cations  $C_{30}H_{40}N_2Z^+$  ( $Z = CH, N, P$ ).

1,3,4,6,7,9-HEXAAZACYCL[3.3.3]AZINE (TRI-S-TRIAZINE)  
 $C_6H_3N_7$

R.S. HOSMANE, M.A. ROSSMAN and N.J. LEONARD, 1982. J. Am. Chem. Soc., **104**, 5497-5499.

Orthorhombic,  $Pbca$ ,  $a = 7.225$ ,  $b = 27.193$ ,  $c = 13.858$  Å,  $Z = 16$ . Cu radiation,  $R = 0.046$  for 1436 reflexions.

The two independent molecules are shown in Fig. 1. One molecule displays anomalous thermal motion and, as a result, bond distances. The other molecule is essentially planar with mean central and peripheral C-N distances of 1.39 and 1.33 Å. In each ring, the spread of the longer internal bonds, C-N-C, meeting at  $\sim 120^\circ$  is compensated by a straightening, via average angles of 116, 129, and  $116^\circ$  of the opposite, apical site containing the shorter bonds, N-C-N.

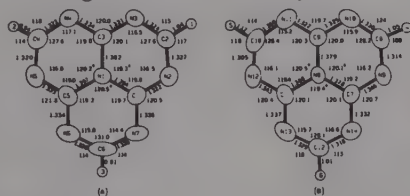


Fig. 1. The two independent tri-s-triazine molecules.

[1,2,4-TRIAZINO][4,5-a]INDOLE  
 $C_{10}H_7N_3$

N.-H. DUNG, D. MAUME and M. ROBBA, 1982. Acta Cryst., **B38**, 1030-1032.

Monoclinic,  $P2_1/b$ ,  $a = 8.557$ ,  $b = 17.336$ ,  $c = 5.800$  Å,  $\gamma = 113.35^\circ$ ,  $D_m = 1.41$ ,  $Z = 4$ . Mo radiation,  $R = 0.035$  for 840 reflexions.

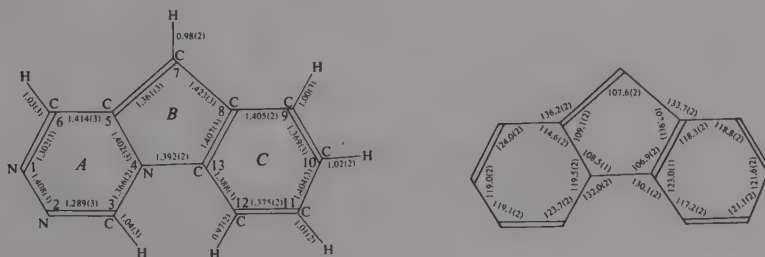
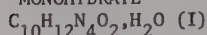


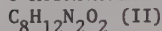
Fig. 1.  $C_{10}H_7N_3$ : bond lengths and angles.

In the molecule (Fig. 1) bond lengths and angles are generally as expected. The packing of the molecules in the crystal is governed by van der Waals interactions.

3,12-DIMETHYL-4,5,10,11-TETRAAZATRICYCLO[6.4.0.0<sup>2,7</sup>]DODECA-3,11-DIENE-6,9-DIONE  
MONOHYDRATE



8-HYDROXYMETHYL-2-METHYL-3,4-DIAZABICYCLO[4.2.0]OCT-2-EN-5-ONE



I.L. KARLE, 1982. *Acta Cryst.*, **B38**, 1019-1022.

I. Monoclinic,  $P2_1/c$ ,  $a = 14.289$ ,  $b = 6.091$ ,  $c = 14.836$  Å,  $\beta = 120.74^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.080$  for 1475 reflexions.

II. Monoclinic,  $P2_1/n$ ,  $a = 7.776$ ,  $b = 11.911$ ,  $c = 9.232$  Å,  $\beta = 102.35^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.064$  for 1046 reflexions.

Dimer I (Fig. 1) has the trans-syn cyclobutane-type configuration with similar bond lengths in the two halves of the molecule. Each of the three rings is planar, with dihedral angles of  $64^\circ$  and  $66^\circ$  between the six-membered ring and the cyclobutyl ring. In the cyclobutane-type addition product II (Fig. 1) the stereochemistry at asymmetric centres C(5), C(6) and C(9) is the same as in dimer I. The dihedral angle between the planes of the six-membered ring and four-membered ring is  $65^\circ$ . Crystals of I contain about 13% of II, disordered among two sites. In both I and II the crystal packing is dominated by N-H...O, O-H...N and O-H...O hydrogen bonds in the range 2.791 to 2.909 Å.

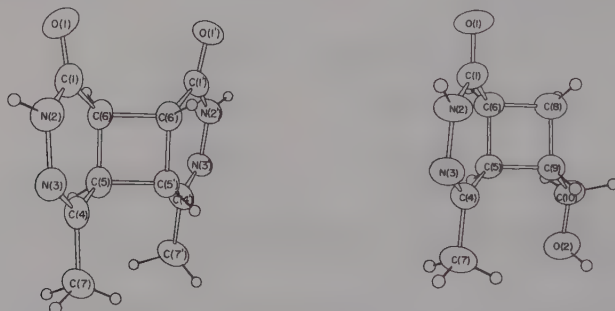
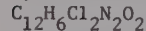


Fig. 1. Perspective views of the  $\text{C}_{10}\text{H}_{12}\text{N}_4\text{O}_2$  (I) (left) and  $\text{C}_8\text{H}_{12}\text{N}_2\text{O}_2$  (II) (right) molecules.

4,5-DICHLORO-9,10-DIAZAPHENANTHRENE N,N'-DIOXIDE



S.N. WHITTLETON and J.D. DUNITZ, 1982. *Acta Cryst.*, **B38**, 2053-2055.

Triclinic,  $P\bar{1}$ ,  $a = 7.419$ ,  $b = 9.652$ ,  $c = 16.662$  Å,  $\alpha = 90.34^\circ$ ,  $\beta = 96.50^\circ$ ,  $\gamma = 106.04^\circ$ ,  $D_m = 1.62$ ,  $Z = 4$ . Mo radiation,  $R = 0.032$  for 3123 reflexions.

The two independent molecules in the asymmetric unit have almost identical structures (Fig. 1). The atoms Cl(1), C(6), C(1), C(7), C(12), Cl(2) form a pronounced spiral with Cl(1)...Cl(2) 3.08 and 3.09 Å. The N-N distances are 1.329(4) and 1.336(4) Å.

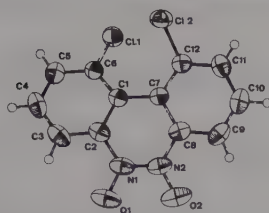


Fig. 1. The  $C_{12}H_6Cl_2N_2O_2$  molecule.

cis-3-ACETYL-2a,7b-DIHYDRO-3H-CYCLOBUT[b]INDOLE  
 $C_{12}H_{11}NO$  (I)

cis-3-PIVALOYL-2a,7b-DIHYDRO-3H-CYCLOBUT[b]INDOLE  
 $C_{15}H_{17}NO$  (II)

M. IKEDA, K. OHNO, M. TAKAHASHI, T. UNO, Y. TAMURA and M. KIDO, 1982. J. Chem. Soc. Perkin I, 741-748.

I. Monoclinic,  $C2/c$ ,  $a = 15.277$ ,  $b = 9.035$ ,  $c = 14.485$  Å,  $\beta = 104.27^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.118$  for 1103 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 8.696$ ,  $b = 15.227$ ,  $c = 9.428$  Å,  $\beta = 102.67^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.108$  for 2123 reflexions.

There are no significant differences between respective bond lengths and angles in I and II (Fig. 1). The methyl group in I is probably disordered. The bond lengths in the cyclobutane ring of II are  $C(1)-C(2)$  1.311,  $C(2)-C(2A)$  1.533,  $C(1)-C(7B)$  1.521 and  $C(2A)-C(7B)$  1.576 Å, and the internal angles are  $C(2A)-C(2)-C(1)$  94.1,  $C(1)-C(7B)-C(2A)$  84.7,  $C(7B)-C(2A)-C(2)$  85.3 and  $C(2)-C(1)-C(7B)$  95.8°. The angles between the cyclobutene and the heterocyclic rings are  $C(1)-C(7B)-C(8)$  113.8 and  $N(3)-C(2A)-C(2)$  116.8°.

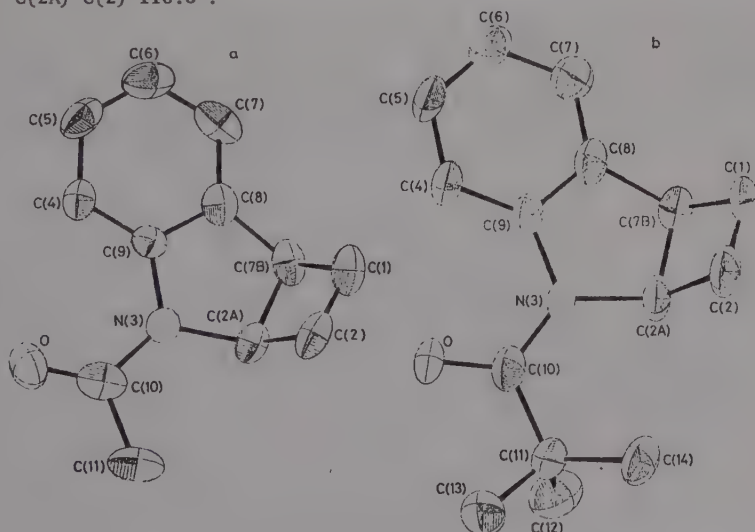


Fig. 1. The molecular structure of  $C_{12}H_{11}NO$  (left) and  $C_{15}H_{17}NO$  (right).



2-CHLORO-8,9,10,11-TETRAHYDRO-6H,11aH-PYRIDAZINO[1,2-a]CINNOLINE-1,4-DIONE  
 $C_{12}H_{13}ClN_2O_2$

A. VEGAS, A. PEREZ-SALAZAR, M. MARTÍNEZ-RIPOLL and S. GARCÍA-BLANCO, 1982. *Acta Cryst.*, B38, 969-971.

Monoclinic,  $P2_1/a$ ,  $a = 13.340$ ,  $b = 18.769$ ,  $c = 4.7181$  Å,  $\beta = 99.53^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.072$  for 1529 reflexions.

In the molecule (Fig. 1) the dihydropyridazine ring has a half-chair conformation with a maximum torsion angle of  $37.4^\circ$  at the N-N bond. The diazaquinone ring has an envelope conformation. The molecules are held together in the crystal by van der Waals forces.

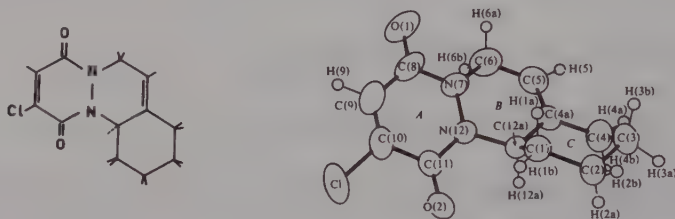


Fig. 1.  $C_{12}H_{13}ClN_2O_2$ : a perspective view of the molecule.

N-METHYL-10-AZABICYCLO[4.3.1]DECANE-8-SPIRO-5'-HYDANTOIN  
 $C_{12}H_{19}N_3O_2$

F. FLORENCIO, P. SMITH-VERDIER and S. GARCÍA-BLANCO, 1982. *Acta Cryst.*, B38, 2089-2091.

Monoclinic,  $P2_1/n$ ,  $a = 6.853$ ,  $b = 11.995$ ,  $c = 14.835$  Å,  $\beta = 90.58^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.042$  for 2621 reflexions.

Ring I (Fig. 1) has a slightly deformed chair conformation with C(3,5') and N(10) displaced -0.523 and 0.665 from the plane of the other ring atoms. Ring II has a boat conformation with N(10), C(7) and C(8) displaced -0.718, -1.153 and -1.200 Å respectively from the C(1), C(5), C(6), C(9) plane. The molecules have approximate mirror symmetry and are linked by hydrogen bonds. Two N(1')-H...O bonds of length 2.886(1) Å are formed between pairs of molecules related by a centre of symmetry while an N(3')-H...N(10) bond of length 2.896(2) Å links molecules into chains along [101].

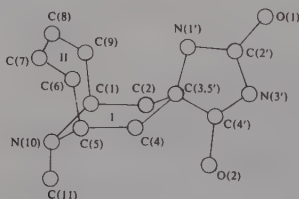


Fig. 1. The  $C_{12}H_{19}N_3O_2$  molecule.

r-4a,c-5a,t-11a-DODECAHYDROPYRIDO[2,1-b]QUINAZOLIN-11-ONE

$C_{12}H_{20}N_2O$  (I)

r-4a,c-5a,c-11a-DODECAHYDROPYRIDO[2,1-b]QUINAZOLIN-11-ONE

$C_{12}H_{20}N_2O$  (II)

F. FULOP, K. SIMON, G. TOTH, I. HERMECZ, Z. MESZAROS and G. BERNATH, 1982. J. Chem. Soc. Perkin I, 2801-2806.

I. Monoclinic,  $P2_1/c$ ,  $a = 6.160$ ,  $b = 9.640$ ,  $c = 19.927$  Å,  $\beta = 95.32^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.087$  for 1423 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 6.390$ ,  $b = 9.975$ ,  $c = 18.229$  Å,  $\beta = 95.69^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.054$  for 2165 reflexions.

In both molecules (Fig. 1) all rings have a chair conformation with the middle ring somewhat distorted especially in II. The lone pair of the bridgehead nitrogen is delocalised towards the carbonyl oxygen in both isomers, again particularly in II. The packing in I is disordered and is described by models A (75% occupancy) and B (25% occupancy) the latter being shown dotted in the top figure. The N(5) hydrogen is axial in both I and II and is intermolecularly hydrogen bonded to O(15). The hydrogen bond is 2.000 in I and 2.18 Å in II, and the respective N-H...O angles are  $158$  and  $160^\circ$ .

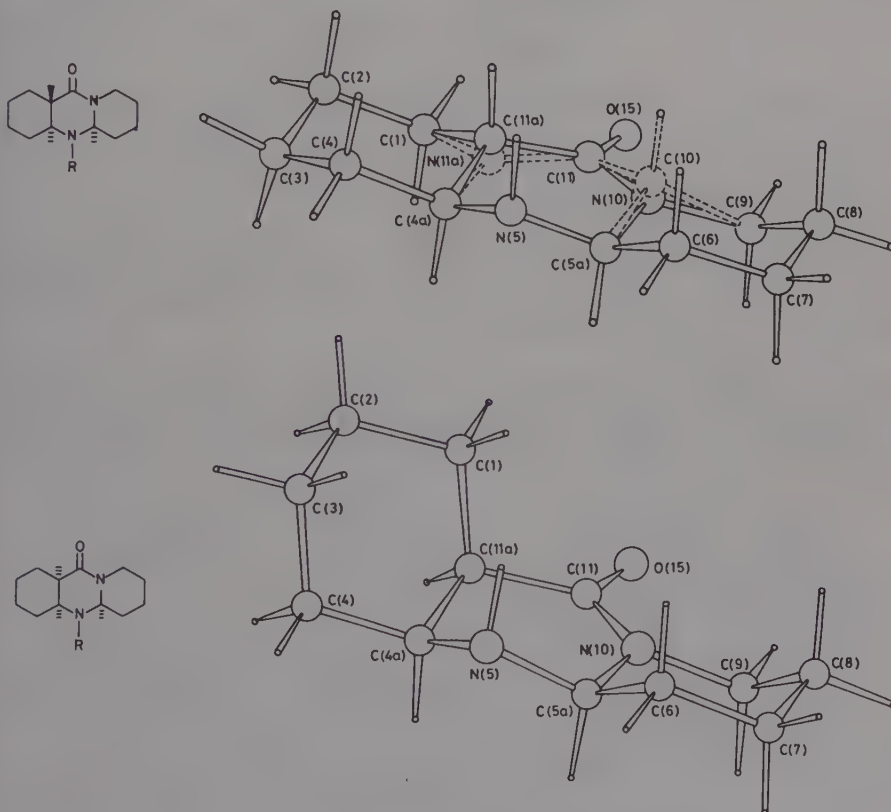


Fig. 1. Molecular skeleton and structure of isomer I (top) and isomer II (bottom) of  $C_{12}H_{20}N_2O$ .

10b,10c-cis-3a,5a,8a,10a-TETRAAZAPERHYDROPYRENE

$C_{12}H_{22}N_4$  (I)

10b,10c-trans-3a,5a,8a,10a-TETRAAZAPERHYDROPYRENE

$C_{12}H_{22}N_4$  (II)

P. GLUZIŃSKI, J.W. KRAJEWSKI, Z. URBANČYK-LIPKOWSKA, J. BLEIDELIS and A. KEMME, 1982. *Acta Cryst.*, B38, 3038-3041.

I. Tetragonal,  $I\bar{4}2d$ ,  $a = 17.176$ ,  $c = 8.447$  Å,  $Z = 8$ . Cu radiation,  $R = 0.068$  for 536 reflexions.

II. Monoclinic,  $P2_1/b$ ,  $a = 5.372$ ,  $b = 8.015$ ,  $c = 14.574$  Å,  $\gamma = 100.78^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.064$  for 706 reflexions.

All rings in both isomers are in chair conformations (Fig. 1). The cis-isomer (I) has twofold crystallographic symmetry and the trans-isomer (II) lies on an inversion centre.

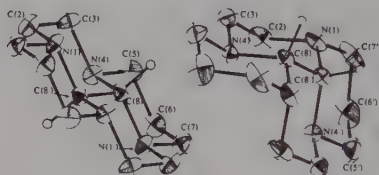


Fig. 1. The trans (left) and cis (right) isomers of  $C_{12}H_{22}N_4$ .

1,4,8,11-TETRAAZATRICYCLO[9.3.1.1<sup>4,8</sup>]HEXADECANE

$C_{12}H_{24}N_4$

E.J. GABE, Y. LE PAGE, L. PRASAD and G.R. WEISMAN, 1982. *Acta Cryst.*, B38, 2752-2754.

Monoclinic,  $P2_1/c$ ,  $a = 9.173$ ,  $b = 10.375$ ,  $c = 6.4530$  Å,  $\beta = 98.68^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.039$  for 972 reflexions (at 115 K).

The molecule (Fig. 1) consists of two asymmetric units related by a centre of symmetry and adopts the expected distorted "diamond lattice" geometry in which each of the two hexahydropyrimidine six-membered rings is a distorted chair with the ethano bridges of the ten-membered ring substituted equatorial/axial (trans) at the bridgehead N atoms.

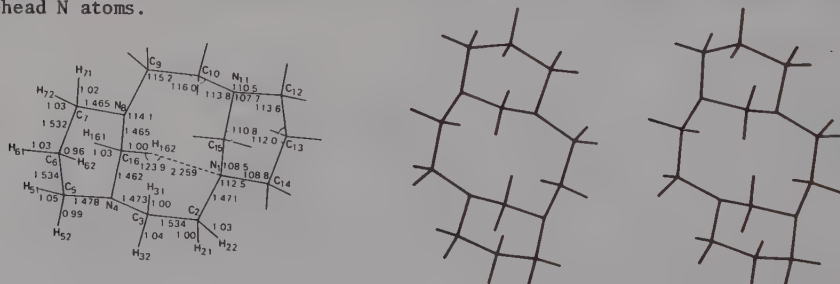
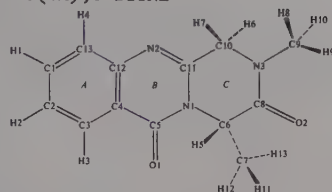


Fig. 1.  $C_{12}H_{24}N_4$ : bond lengths and angles and a stereoview of the molecule.

## (4S)-2,4-DIMETHYL-1,2-DIHYDROPIRAZINO[2,1-b]QUINAZOLINE-3(4H),6-DIONE

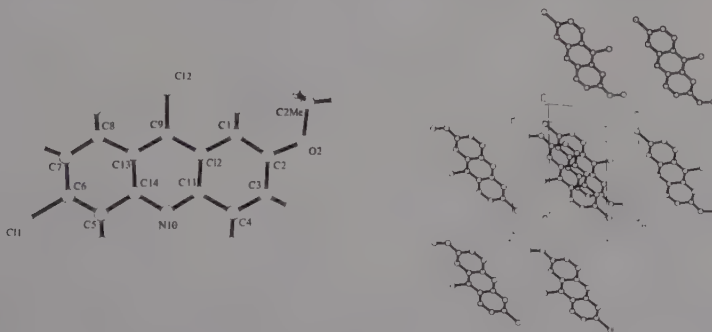
 $C_{13}H_{13}N_3O_2$ K. SUGUNA, S. RAMAKUMAR and S. RAJAPPA, 1982. *Acta Cryst.*, **B38**, 1654-1656.Orthorhombic,  $P2_12_12_1$ ,  $a = 17.443$ ,  $b = 11.650$ ,  $c = 5.784$  Å,  $D_m = 1.456$ ,  $Z = 4$ . Cu radiation,  $R = 0.040$  for 1358 reflexions.

The N-methylated, *cis* peptide group is non-planar; the torsion angle about the peptide bond is  $-6.1(4)^\circ$  and the peptide bond length is  $1.337(3)$  Å. N(3) is  $0.128$  Å from the C(9), C(10), C(8) plane and C(8) is  $0.006$  Å from the N(3), O(2), C(6) plane. C(7) is axial. Rings A and B are planar while C is a flattened boat. The structure is stabilised by C-H...O interactions with C(7)...O(1)  $3.283(3)$  and C(3)...O(2)  $3.483(3)$  Å; the C-H...O angles are  $137(3)$  and  $162(3)^\circ$  respectively.

## 6,9-DICHLORO-2-METHOXYACRIDINE

 $C_{14}H_9Cl_2NO$ S. NEIDLE, 1982. *Acta Cryst.*, **B38**, 159-162.Triclinic,  $P\bar{1}$ ,  $a = 7.735$ ,  $b = 8.651$ ,  $c = 10.344$  Å,  $\alpha = 95.63$ ,  $\beta = 101.80$ ,  $\gamma = 113.25^\circ$ ,  $D_m = 1.52$ ,  $Z = 2$ . Cu radiation,  $R = 0.040$  for 1999 reflexions.

The structure consists of centrosymmetrically related, stacked molecules (Fig. 1). The acridine nucleus is slightly buckled, and does not show mirror symmetry along the C(9)-N(10) line. The addition of the Cl atom at C(9) has produced significant shortening in adjacent bonds, compared to acridines with a Cl atom at C(6) alone.

Fig. 1.  $C_{14}H_9Cl_2NO$ : molecular structure and crystal structure.

## 4,5-DIMETHYL-9,10-DIAZAPHENANTHRENE N,N'-DIOXIDE

 $C_{14}H_{12}N_2O_2$ S.N. WHITTLETON and J.D. DUNITZ, 1982. *Acta Cryst.*, **B38**, 2052-2053.Monoclinic,  $P2_1/c$ ,  $a = 8.420$ ,  $b = 15.632$ ,  $c = 9.213$  Å,  $\beta = 112.05^\circ$ ,  $D_m = 1.42$ ,  $Z = 4$ . Mo radiation,  $R = 0.032$  for 1571 reflexions.

The molecule (Fig. 1) has approximate twofold symmetry, with N-N 1.339(2) Å. There is distortion in the central portion to relieve steric repulsion between the 4,5 dimethyl groups (C(100)...C(200) is 3.03 Å with atoms C(100)-C(6)-C(1)-C(7)-C(12)-C(200) forming a pronounced spiral). The diazine ring has an almost perfect twist conformation.

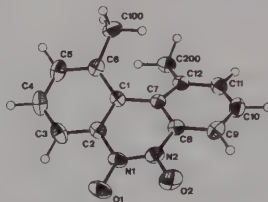


Fig. 1. The  $C_{14}H_{12}N_2O_2$  molecule.

10 $\beta$ ,11 $\beta$ -DIHYDROXY-11 $\alpha$ -METHYL-1,3,4,6,6 $\alpha\beta$ ,7,8,9,10 $\alpha$ ,10 $\alpha\alpha$ ,11,11 $\alpha\beta$ -DODECAHYDRO-2H-BENZO-[b]QUINOLIZINE  
 $C_{14}H_{25}NO_2$

R. BRAMBILLA, R. FRIARY, A. GANGULY, M.S. PUAR, J.G. TOPLISS, R. WATKINS and A.T. McPHAIL, 1982. J. Org. Chem., 47, 4137-4145.

Monoclinic,  $P2_1/c$ ,  $a = 10.437$ ,  $b = 8.663$ ,  $c = 16.720$  Å,  $\beta = 114.31^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.060$  for 1615 reflexions.

The analysis confirmed the assignment of the relative stereochemistry at C6 $\alpha$  and C11 as shown in Fig. 1, and established that the solid state conformation was all-chair.

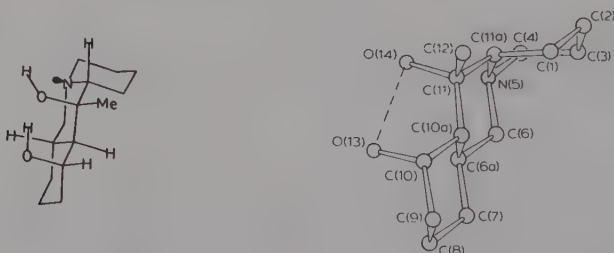


Fig. 1. A view of  $C_{14}H_{25}NO_2$ .

1-(2-AMINO-4-PYRIMIDINYL)- $\beta$ -CARBULINE HYDRATE  
 $C_{15}H_{11}N_5 \cdot H_2O$

Y. YOKOMORI, K. SEKIDO, T.-S. WU, H.-J. TIEN and S. HIROKAWA, 1982. Bull. Chem. Soc. Jpn., 55, 2236-2238.

Monoclinic,  $P2_1/c$ ,  $a = 20.699$ ,  $b = 4.453$ ,  $c = 15.720$  Å,  $\beta = 112.16^\circ$ ,  $D_m = 1.396$ ,  $Z = 4$ . Mo radiation,  $R = 0.109$  for 1415 reflexions.

The analysis establishes the structure as shown in Fig. 1. Molecular dimensions are normal.

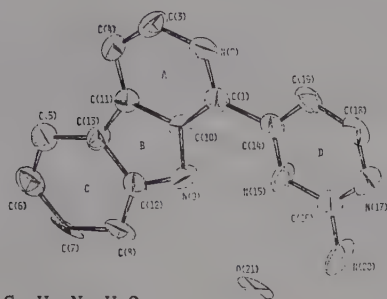


Fig. 1. A view of  $C_{15}H_{11}N_5 \cdot H_2O$ .

4-ETHOXYINDENO[1,2-d]AZEPINE  
 $C_{15}H_{13}NO$

M. MIYAKE, M. KIMURA, S. MOROSAWA, S. KASHINO and M. HAISA, 1982. *Acta Cryst.*, **B38**, 2080-2082.

Monoclinic,  $P2_1/a$ ,  $a = 23.752$ ,  $b = 5.834$ ,  $c = 8.485$  Å,  $\beta = 92.22^\circ$ ,  $D_m = 1.268$ ,  $Z = 4$ . Cu radiation,  $R = 0.044$  for 1650 reflexions.

The indenoazepine ring (Fig. 1) is planar within  $0.102(2)$  Å. The azepine ring has a twist-boat conformation while the five-membered ring is slightly puckered into a half-chair conformation. Bond lengths are in agreement with those calculated by the LCAO-SCF-MO method. Molecules are arranged in sheets parallel to (100) with sheets stacked along  $a$ .

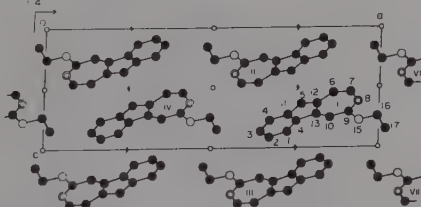


Fig. 1. The 4-ethoxyindeno[1,2-d]azepine structure projected along  $b$ .

5,6-DIHYDRO-8-METHYL-4H-PYRAZINO[3,2,1-j,k]CARBAZOLE HYDROCHLORIDE HYDRATE  
 $C_{15}H_{15}ClN_2 \cdot 5(H_2O)$  (I)

2,4,5,6-TETRAHYDRO-8-METHYL-1H-PYRAZINO[3,2,1-j,k]CARBAZOLE HYDROCHLORIDE HYDRATE  
 $C_{15}H_{17}ClN_2 \cdot 2(H_2O)$  (II)

2,3,3a,4,5,6-HEXAHYDRO-8-METHYL-1H-PYRAZINO[3,2,1-j,k]CARBAZOLE HYDROCHLORIDE HYDRATE  
 $C_{15}H_{19}ClN_2 \cdot 2(H_2O)$  (III)

H.A. KARAPETYAN, Yu.T. STRUCHKOV and G.G. DVORYANTSEVA, 1982. *Cryst. Struct. Comm.*, **11**, 1441-1458.

I. Orthorhombic,  $Pn2_1a$ ,  $a = 17.042$ ,  $b = 6.895$ ,  $c = 15.412$  Å,  $Z = 4$ . Mo radiation,  $R = 0.071$  for 974 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 7.379$ ,  $b = 10.307$ ,  $c = 10.980$  Å,  $\alpha = 66.18$ ,  $\beta = 78.02$ ,  $\gamma = 85.39^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.060$  for 1051 reflexions.

III. Monoclinic,  $P2_1/a$ ,  $a = 16.232$ ,  $b = 5.757$ ,  $c = 17.862$  Å,  $\beta = 114.68^\circ$ ,  $Z = 4$ .



Mo radiation,  $R = 0.038$  for 2543 reflexions.

The indole fragments of the cations (Fig. 1) are essentially planar; the cyclohexene rings of I and II have symmetrical sofa conformations, whereas in III a half-chair form is found. The six-membered heterocyclic ring is planar in I, has a flattened half-chair conformation in II, and has a severely distorted chain conformation in III.

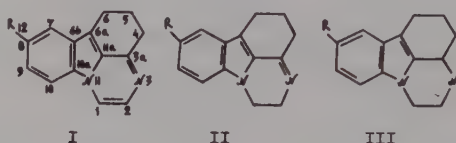


Fig. 1. The parent carbazoles of I, II, and III.

5-CHLORO-3-(2-CHLOROPHENYL)-1,3-DIHYDRO-3-HYDROXYAZIRINO[1,2-b]ISOQUINOLIN-8(8aH)-ONE  
 $C_{16}H_{11}Cl_2NO_2$

E.J. TRYBULSKI, E. REEDER, J.F. BLOUNT, A. WALSER and R.I. FRYER, 1982. *J. Org. Chem.*, **47**, 2441-2447.

Triclinic,  $P\bar{1}$ ,  $a = 8.145$ ,  $b = 9.737$ ,  $c = 10.159$  Å,  $\alpha = 98.48$ ,  $\beta = 102.53$ ,  $\gamma = 111.77^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.056$  for 1515 reflexions.

X-ray analysis was used to confirm the structure shown in Fig. 1.

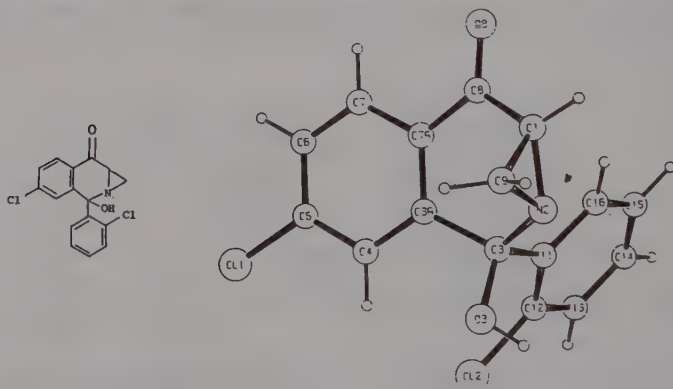


Fig. 1. The  $C_{16}H_{11}Cl_2NO_2$  molecule.

3,5,5,10,10,12-HEXAMETHYL-3,12-DIAZATRICYCLO[6.4.0.0<sup>2,7</sup>]DODECANE-4,6,9,11-TETRAONE  
 $C_{16}H_{22}N_2O_4$  (I)

1,2,3,5,5,10,10,12-OCTAMETHYL-3,12-DIAZATRICYCLO[6.4.0.0<sup>2,7</sup>]DODECANE-4,6,9,11-TETRAONE  
 $C_{18}H_{26}N_2O_4$  (II)

I.L. KARLE, 1982. *Acta Cryst.*, **B38**, 1016-1018.

I. Monoclinic,  $P2_1/c$ ,  $a = 6.370$ ,  $b = 13.989$ ,  $c = 17.598$  Å,  $\beta = 100.56^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.059$  for 1901 reflexions.

II. Monoclinic,  $P2_1/a$ ,  $a = 12.245$ ,  $b = 11.929$ ,  $c = 12.467$  Å,  $\beta = 108.92^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.074$  for 2180 reflexions.

Both dimers I and II (Fig. 1) have the trans-syn configuration. Bond lengths and angles are similar not only in each half of each dimer but this similarity is also maintained between dimers I and II. However, the C(6)-C(6') lengths (1.579 Å in I and 1.613 Å in II) differ; this increase in length can be related to the presence of methyl groups on C(6) and C(6'). The packing of the molecules in the crystals is dominated by van der Waals contacts between methyl groups.

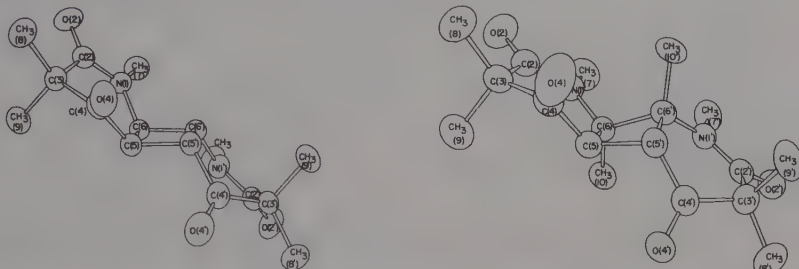


Fig. 1. Perspective views of the  $C_{16}H_{22}N_2O_4$  (left) and  $C_{18}H_{26}N_2O_4$  (right) molecules.

(6S,13bR)-1,2,3,5,6,13b-HEXAHYDRO-6-ISOPROPYL-8H-PYRROLO[1',2':1,2]PYRAZINO[3,4-b]-QUINAZOLINE-5,8-DIONE

$C_{17}H_{19}N_3O_2$

K. SUGUNA, S. RAMAKUMAR and S. RAJAPPA, 1982. Acta Cryst., B38, 2304-2306.

Monoclinic,  $P2_1$ ,  $a = 5.382$ ,  $b = 17.534$ ,  $c = 8.198$  Å,  $\beta = 100.46^\circ$ ,  $D_m = 1.323$ ,  $Z = 2$ . Cu radiation,  $R = 0.052$  for 1284 reflexions.

In the molecule (Fig. 1) the proline-containing cis-peptide unit which forms part of a six-membered ring deviates from perfect planarity. The torsion angle about the peptide bond is  $3.0(5)^\circ$  and the peptide bond length is  $1.313(5)$  Å. The conformation of the proline ring is  $C_s$ -C $\beta$ -endo. The crystal structure is stabilised by C-H...O interactions.

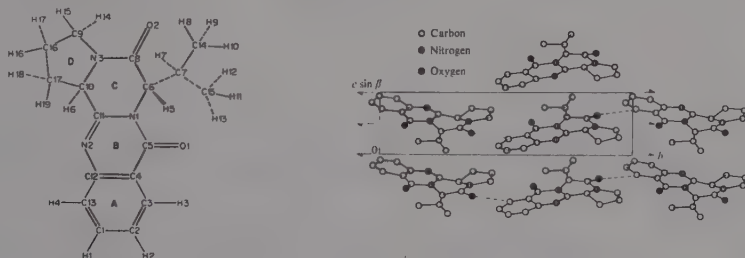


Fig. 1.  $C_{17}H_{19}N_3O_2$ : views of the molecule and of the packing of the molecules along a.

METHYL-1,2,5,6,7,7a,8,8a-OCTAHYDRO-8-(METHOXYCARBONYL)-3-(TRIFLUOROACETYL)CYCLOPENTAN[b]PYRROLIZINE-8-ACETATE

$C_{17}H_{20}F_3NO_5$

W. VERBOOM, D.N. REINHOUT, S. HARKEMA and G.J. VAN HUMMEL, 1982. J. Org. Chem., 47, 3339-3342.

Triclinic,  $P\bar{1}$ ,  $a = 13.889$ ,  $b = 10.462$ ,  $c = 6.801$  Å,  $\alpha = 73.77^\circ$ ,  $\beta = 75.10^\circ$ ,  $\gamma = 73.42^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.055$  for 2648 reflexions.

The analysis establishes the structure of this reaction product to be as shown in Fig. 1.

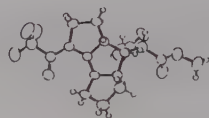
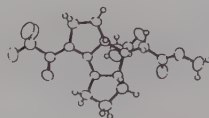
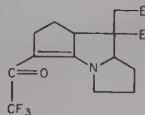


Fig. 1. The C<sub>17</sub>H<sub>20</sub>F<sub>3</sub>NO<sub>5</sub> molecule (E = CO<sub>2</sub>CH<sub>3</sub>).

15H-DIBENZO[3,4:5,6]AZEPINO[2,1-f]PURINE  
C<sub>18</sub>H<sub>12</sub>N<sub>9</sub>

I. AGRANAT, M. RABINOVITZ, C.-P. TANG and W.-C. SHAW, 1982. J. Org. Chem., 47, 1578-1579.

Orthorhombic, Pbca, a = 18.804, b = 12.220, c = 12.129 Å, Z = 8. Cu radiation, R = 0.059 for 2547 reflexions.

The analysis established the structure shown in Fig. 1.

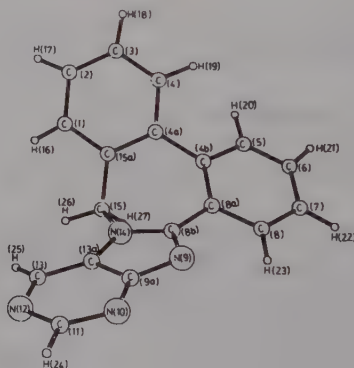
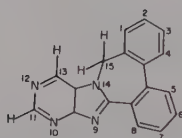
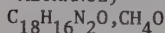


Fig. 1. The C<sub>18</sub>H<sub>12</sub>N<sub>9</sub> molecule.

9-METHOXY-5,11-DIMETHYL-6H-PYRIDO[3,4-b]CARBAZOLE METHANOL (9-METHOXYELLIPTICINE METHANOL)



C. COURSEILLE, B. BUSETTA and M. HOSPITAL, 1982. Cryst. Struct. Comm., 11, 893-896.

Orthorhombic, P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, a = 17.922, b = 19.430, c = 4.534 Å, Z = 4. Cu radiation, R = 0.08 for 1045 reflexions.

The ellipticine ring system is planar. The stacking of the molecules is shown in Fig. 1; the interplanar separation is 3.49 Å. Molecules are linked by hydrogen bonds involving methanol.

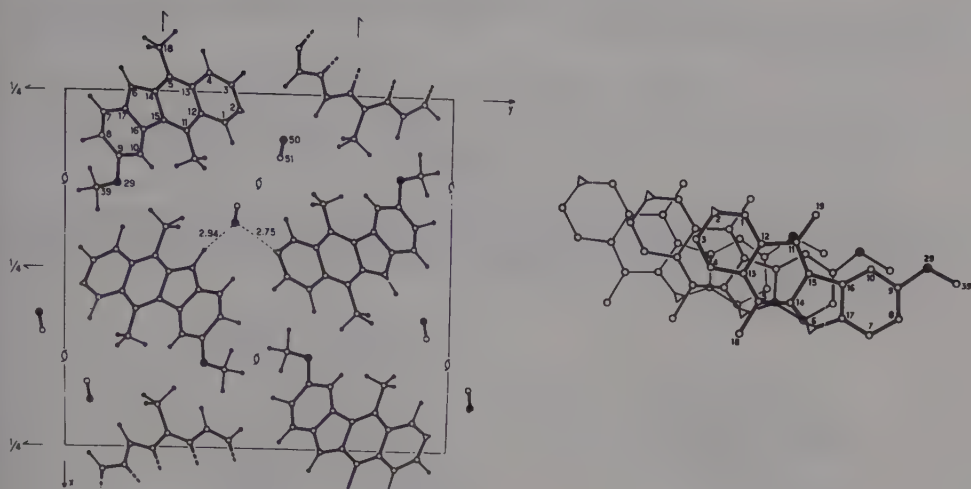


Fig. 1. The molecular packing and stacking of  $C_{18}H_{16}N_2O, CH_4O$ .

9-HYDROXY-5,11-DIMETHYL-6N-METHYL-PYRIDO[3,4-b]CARBAZOLIUM CHLORIDE (6N-METHYL-9-HYDROXYELLIPTICINIUM CHLORIDE)  
 $C_{18}H_{17}ClN_2$

C. COURSEILLE, B. BUSETTA and M. HOSPITAL, 1982. Cryst. Struct. Comm., 11, 897-900.

Triclinic,  $P\bar{1}$ ,  $a = 10.718$ ,  $b = 9.186$ ,  $c = 7.964$  Å,  $\alpha = 88.84$ ,  $\beta = 91.74$ ,  $\gamma = 107.17^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.07$  for 1914 reflexions.

The ellipticine system is nearly planar and the cations are stacked along  $c$  with interplanar separations of 3.45 and 3.40 Å (Fig. 1). Cations are linked by hydrogen bonds involving the chloride ions and pyridine nitrogen.

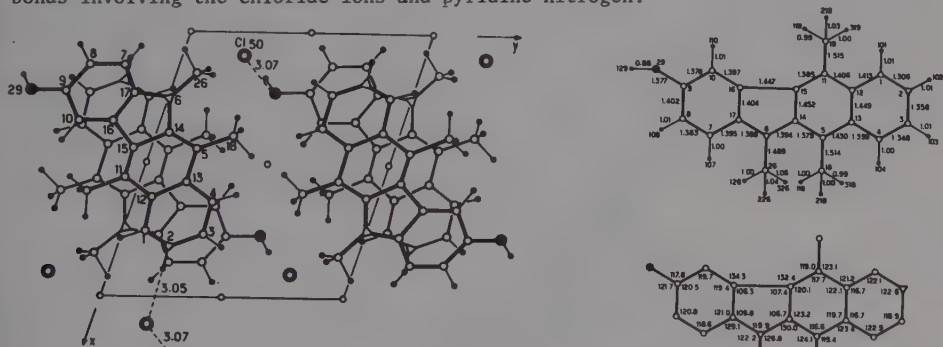


Fig. 1. Molecular packing of  $C_{18}H_{17}ClN_2$  and dimensions.

9-METHOXY-5,11-DIMETHYL-6H-PYRIDO[3,4-b]CARBAZOLIUM CHLORIDE MONOHYDRATE (9-METHOXY-ELLIPTICINIUM CHLORIDE MONOHYDRATE)  
 $C_{18}H_{17}ClN_2O \cdot H_2O$

C. COURSEILLE, B. BUSETTA and M. HOSPITAL, 1982. Cryst. Struct. Comm., 11, 889-892.

Triclinic,  $P\bar{1}$ ,  $a = 14.504$ ,  $b = 12.169$ ,  $c = 4.806$  Å,  $\alpha = 108.45$ ,  $\beta = 92.80$ ,  $\gamma = 93.22^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.07$  for 1740 reflexions.

The ellipticine ring system is planar and the main feature of the structure is the stacking along the c-direction (Fig. 1) with an interplanar spacing of 3.33 Å. Along the b-direction molecules are linked by hydrogen bonds involving the water molecule and chloride ion.

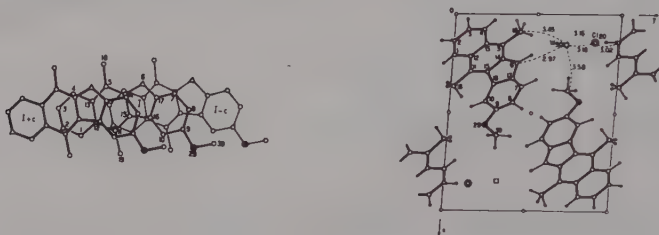


Fig. 1. Stacking of  $C_{18}H_{17}ClN_2O \cdot H_2O$  and a projection along c.

8-CHLORO-11-(4-METHYL-1-PIPERAZINYL)-5H-DIBENZO[b,c][1,4]DIAZEPINE DIHYDROBROMIDE (CLOZAPINE DIHYDROBROMIDE)

$C_{18}H_{21}BrClN_4$

$C_{18}H_{19}ClN_4 \cdot 2HBr$

J.P. FILLERS and S.W. HAWKINSON, 1982. Acta Cryst., B38, 1750-1753.

Monoclinic,  $P2_1/c$ ,  $a = 10.047$ ,  $b = 11.775$ ,  $c = 16.711$  Å,  $\beta = 99.94^\circ$ ,  $D_m = 1.64$ ,  $Z = 4$ . Mo radiation,  $R = 0.044$  for 4498 reflexions.

The molecular structure (Fig. 1) is similar to that of the free base (1) except at sites of protonation; bond lengths about N(10) and N(19) are increased and the C(13)-N(10)-C(11) angle is  $128.6(3)^\circ$  compared to  $121.4(2)^\circ$ . The angle between the normals to the benzene rings is  $129^\circ$ . The seven-membered ring and the piperazine ring have boat and chair conformations respectively with the N(10)-C(11)-N(16)-C(21) torsion angle  $17.5^\circ$ . Anions and cations are linked by a network of N-H...Br hydrogen bonds ( $3.243(3)$ - $3.370(3)$  Å).

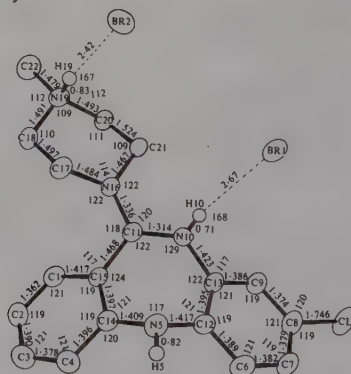
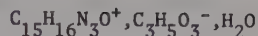
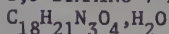


Fig. 1. Bond lengths and angles in clozapine dihydrobromide.

1. Structure Reports, 42B, 223.

## 3,9-DIAMINO-7-ETHOXYACRIDINE LACTATE MONOHYDRATE SALT



S. NEIDLE and A. AGGARWAL, 1982. Acta Cryst., B38, 2420-2424.

Triclinic,  $\bar{P}1$ ,  $a = 7.912$ ,  $b = 10.016$ ,  $c = 12.246$  Å,  $\alpha = 107.75$ ,  $\beta = 103.08$ ,  $\gamma = 94.47^\circ$ ,  $D_m = 1.35$ ,  $Z = 2$ . Cu radiation,  $R = 0.063$  for 2149 reflexions.

The molecular geometry reflects the 3,9-diamino substitution of the planar acridine ring in which the central N(10) atom is protonated rather than the terminal amino groups (Fig. 1). The crystal structure is extensively hydrogen-bonded with interactions involving anions, acridine substituents and water molecules.

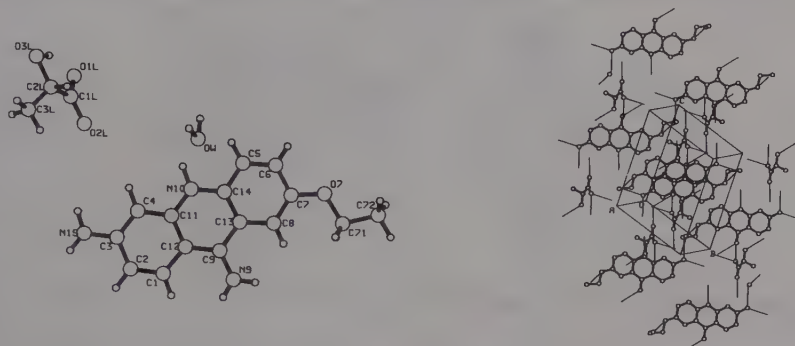
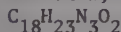


Fig. 1.  $\text{C}_{15}\text{H}_{16}\text{N}_3\text{O}^+, \text{C}_3\text{H}_5\text{O}_3^-, \text{H}_2\text{O}$ : molecular structure and crystal structure.

## METHYL 1,2-BIS(DIMETHYLAMINO)-4-METHYL-1H-PYRROLO[1,2-a]INDOLE-3-CARBOXYLATE



G. FERGUSON, B. KAITNER, W.B. WHALLEY, D.A. TAYLOR and J.R. LIDDELL, 1982. Acta Cryst., B38, 2758-2760.

Orthorhombic,  $Pbca$ ,  $a = 8.349$ ,  $b = 23.465$ ,  $c = 17.387$  Å,  $Z = 8$ . Mo radiation,  $R = 0.059$  for 786 reflexions.

The molecule (Fig. 1) shows signs of intramolecular overcrowding. The benzene ring is close to planar, but the five-membered ring, N(1),C(8)-C(11) adopts a slight envelope conformation with C(9) at the flap. As a result the molecule is slightly folded and there is a  $12.5^\circ$  angle between the benzene and N(1),C(8)-C(11) ring planes. Molecular dimensions are generally as expected and the molecules are separated in the crystal by normal van der Waals distances.

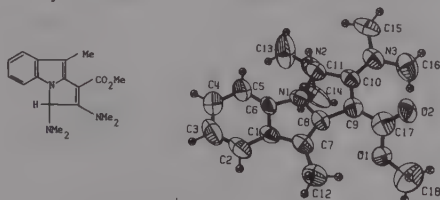


Fig. 1.  $\text{C}_{18}\text{H}_{23}\text{N}_3\text{O}_2$ : structural formula and perspective view of the molecule.



2-METHOXYCARBONYL-3-METHYL-1,2,3,4-TETRAHYDRONAPHTHO[1,2-b]N-PHENYLAZIRIDINE  
 $C_{19}H_{19}NO_2$

B. TINANT, J.P. DECLERCQ, G. GERMAIN and M. VAN MEERSSCHE, 1982. Cryst. Struct. Comm., 11, 1405-1410.

Triclinic,  $P\bar{1}$ ,  $a = 10.396$ ,  $b = 10.274$ ,  $c = 8.085$  Å,  $\alpha = 111.55$ ,  $\beta = 91.60$ ,  $\gamma = 84.37^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.039$  for 1834 reflexions.

The cyclohexene ring of the three fused rings (Fig. 1) has what is described as a "chair-twist-boat transition form". The methoxycarbonyl moiety is approximately coplanar with the tetrahydronaphtho system.

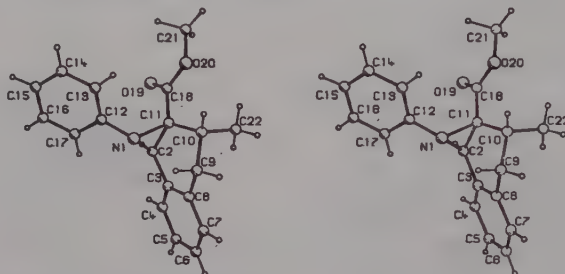


Fig. 1. A stereoview of  $C_{19}H_{19}NO_2$ .

2-METHOXYCARBONYL-4-METHYL-1,2,3,4-TETRAHYDRONAPHTHO[1,2-b]N-PHENYLAZIRIDINE  
 $C_{19}H_{19}NO_2$

B. TINANT, J.P. DECLERCQ, G. GERMAIN and M. VAN MEERSSCHE, 1982. Cryst. Struct. Comm., 11, 1411-1416.

Monoclinic,  $P2_1/a$ ,  $a = 11.877$ ,  $b = 8.657$ ,  $c = 15.572$  Å,  $\beta = 100.96^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.053$  for 1653 reflexions.

The cyclohexene ring (Fig. 1) adopts a "chair-twist-boat transition form". The 4-methyl substituent lies approximately in the mean plane defined by the methoxycarbonyl and tetrahydronaphtho moiety (pseudo-equatorial position) while the aziridine ring and 4-hydrogen atom (H(C9)) are both on the same side of this plane.

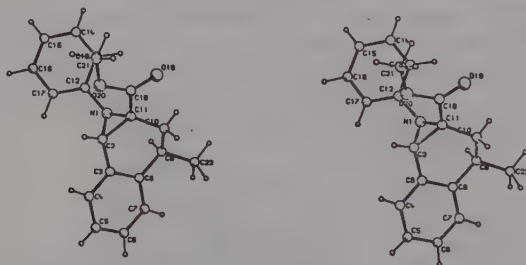


Fig. 1. A stereoview of  $C_{19}H_{19}NO_2$ .

2,2'-(2,3,5,10-TETRAHYDRO-5,10-DIMETHYLPHENAZINE-2,3-DIYLIDENE) BIS (PROPANEDINITRILE)  
 $C_{20}H_{12}N_6$

K. DIETZ, H.J. KELLER, D. NÜTTE and D. WEHE, 1982. J. Am. Chem. Soc., 104, 7581-7585.

Monoclinic,  $C2/c$ ,  $a = 9.674$ ,  $b = 19.411$ ,  $c = 9.603$  Å,  $\beta = 113.20^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.076$  for 1200 reflexions.

The molecule (Fig. 1) lies on a twofold axis. Rings A and B are planar while the C ring (bearing the two  $C(CN)_2$  substituents) is significantly twisted. The molecules form regular stacks along  $c$  with interplanar distances of  $4.75(2)$  Å. Bond distances ( $\sigma$  0.006-0.012 Å) are normal.

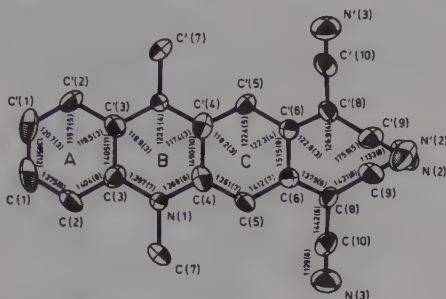


Fig. 1. Geometry of  $C_{20}H_{12}N_6$ .

2-[2-(DIETHYLAMINO)ETHYL]-2H-[1]BENZOTHIOPYRANO[4,3,2-cd]INDAZOLE-5-METHANOL MONO-HYDRATE  
 $C_{20}H_{23}N_3OS \cdot H_2O$

C.H. WEI, 1982. Acta Cryst., B38, 548-553.

Rhombohedral,  $R\bar{3}$ ,  $a = b = 36.685$ ,  $c = 7.7432$  Å,  $D_m = 1.25$ ,  $Z = 18$ . Cu radiation,  $R = 0.042$  for 2804 reflexions.

The structure contains neutral molecules of the base (Fig. 1) linked by hydrogen bonds to water molecules. Each hydroxyl O and N(3) atom is a hydrogen bond acceptor of hydrogen atoms from different water molecules with  $O \cdots N$  2.786 and 2.977 Å and  $O \cdots O$  2.851 Å. The terminal N(2) atom is not protonated but is hydrogen-bonded to the hydroxyl group of a neighbouring, screw-triad-related molecule forming left- and right-handed coils around these axes in the  $c$  direction. The fused four-ring system is virtually planar, with some stacking interactions between pairs of centrosymmetrically related molecules.

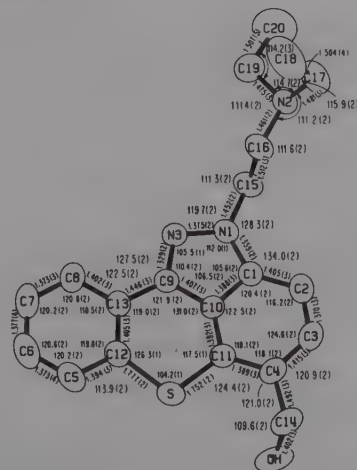


Fig. 1. Bond lengths and angles in the  $C_{20}H_{23}N_3OS$  molecule.

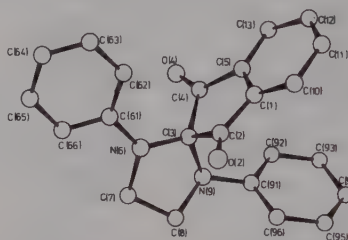
## 1,3-DIPHENYLSPIRO[IMIDAZOLIDINE-2,2'-INDAN]-1',3'-DIONE

 $C_{23}H_{18}N_2O_2$ 

W.S. SHELDRICK, A. SCHÖNBERG and E. SINGER, 1982. Acta Cryst., B38, 1355-1357.

Triclinic,  $P\bar{1}$ ,  $a = 9.225$ ,  $b = 12.982$ ,  $c = 8.286$  Å,  $\alpha = 102.11$ ,  $\beta = 108.93$ ,  $\gamma = 95.53^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.047$  for 2394 reflexions.

The molecule (Fig. 1) contains neither an approximate twofold rotation axis nor an approximate mirror plane. One of the N atoms displays a trigonal-planar the other a pyramidal coordination geometry. The tetrahedral geometry of the spiro C atom is significantly flattened (angles at C(3)  $101.8(2)$  to  $117.1(2)^\circ$ ).

Fig. 1.  $C_{23}H_{18}N_2O_2$ : a perspective view of the molecule.

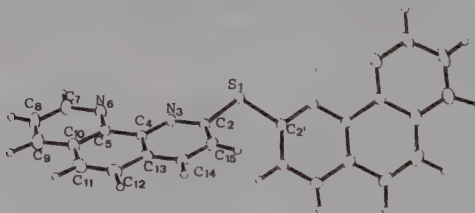
## 2,2'-THIO-BIS-1,10-PHENANTHROLINE CHLOROFORM SOLVATE

 $C_{24}H_{14}N_4S \cdot 2CHCl_3$ 

U. HAUSER, M. HUNZIKER and G. RIHS, 1982. Cryst. Struct. Comm., 11, 1515-1518.

Orthorhombic,  $Pccn$ ,  $a = 11.889$ ,  $b = 20.397$ ,  $c = 11.628$  Å,  $Z = 4$ . Mo radiation,  $R = 0.071$  for 1989 reflexions.

The molecule has the sulphur atom on a crystallographic twofold axis (Fig. 1) with C-S  $1.786(5)$  Å, C-S-C  $103.4(4)^\circ$ . The sulphur atom is  $0.26$  Å from the phenanthroline plane. The chloroform molecules are disordered.

Fig. 1. A view of  $C_{24}H_{14}N_4S$ .

## SPIRO[ISOINDOLINE-4-(3,4,5,6-TETRAHYDRO-2,2,7,7-TETRAMETHYL-3-CARBOMETHOXY)-3'-(2',2',5',5'-TETRAMETHYL-4'-METHYLENOCARBOMETHOXY)PYRROLE] DIHYDRATE

 $C_{24}H_{38}N_2O_4 \cdot 2H_2O$ 

G. BOCELLI, 1982. Cryst. Struct. Comm., 11, 2035-2041.

Monoclinic,  $Cc$ ,  $a = 18.943$ ,  $b = 11.318$ ,  $c = 24.376$  Å,  $\beta = 94.5^\circ$ ,  $[Z = 8]$ . Cu radiation,  $R = 0.080$  for 2949 reflexions.

The two independent molecules show some differences in their stereochemistry, particularly in the planarity of some fragments. The two uncondensed pyrrole rings assume different conformations (twist for one, near envelope in the second). The

two six-membered rings show both a conformation near half-boat and the condensed pyrrole rings are both twist. Bond lengths and angles are as anticipated. A view of one molecule is in Fig. 1.

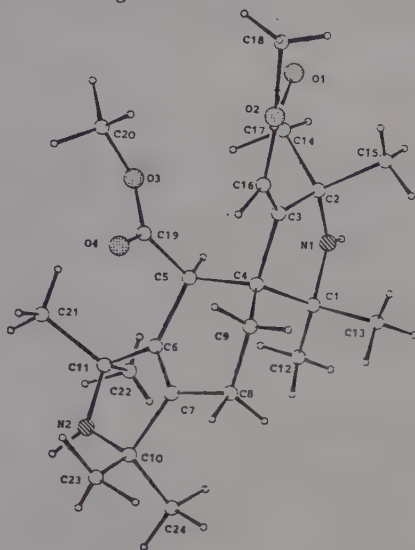
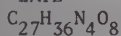


Fig. 1. A view of one of the  $C_{24}H_{38}N_2O_4$  molecules.

TETRAMETHYL 1,1a,8a,9,9a,9b,9c,9d-OCTAHYDRO-1,1,9,9-TETRAMETHYL-5-(2-METHYL-1-PROPENYL) DICYCLOPROP(D,D') IMIDAZO(1,5-6:3,4-B') DIPYRIDAZINE-2,8,9b,9c-TETRACARBOXY-LATE



F.-X. HUBER, J. SAUER, W.S. McDONALD and H. NOTH, 1982. Chem. Ber., 115, 444-451.

Triclinic,  $P\bar{1}$ ,  $a = 10.087$ ,  $b = 15.321$ ,  $c = 9.990$  Å,  $\alpha = 91.95$ ,  $\beta = 111.02$ ,  $\gamma = 100.06^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.037$  for 2736 reflexions.

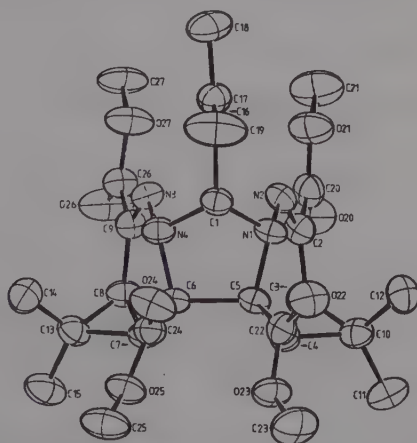


Fig. 1. The structure of  $C_{27}H_{36}N_4O_8$ .

Except for the elongated C(5)-C(6) bond (1.652(3) Å) the bond lengths in the molecule (Fig. 1) have normal values. The central  $C_3N_2$  five-membered ring adopts an

envelope conformation with C(1) at the flap, 0.70 Å above the mean plane through N(1), C(5), C(6), N(4). The mean plane through the dimethylvinyl group on C(1) is nearly perpendicular to the five-membered ring mean plane while the dihedral angles between the latter and the six-membered ring mean planes are 67.9 and 71.1°.

### 5,13-DIAZAPYRANTHRENE (FLAVANTHRENE)

$C_{28}H_{14}N_2$

K. TORIUMI, K. KOYANO, N. SATO, H. TAKAYA, T. ITO and H. INOKUCHI, 1982. *Acta Cryst.*, B38, 959-961.

Monoclinic,  $P2_1/a$ ,  $a = 16.179$ ,  $b = 3.7996$ ,  $c = 15.818$  Å,  $\beta = 119.192^\circ$ ,  $D_m = 1.47$ ,  $Z = 2$ . Cu radiation,  $R = 0.041$  for 1445 reflexions.

The molecule (Fig. 1) is planar with C-C distances in the range 1.347-1.450 Å. The interplanar spacing between molecules is 3.4711 Å. The proportion of overlapped area of the molecular plane is about 54%.

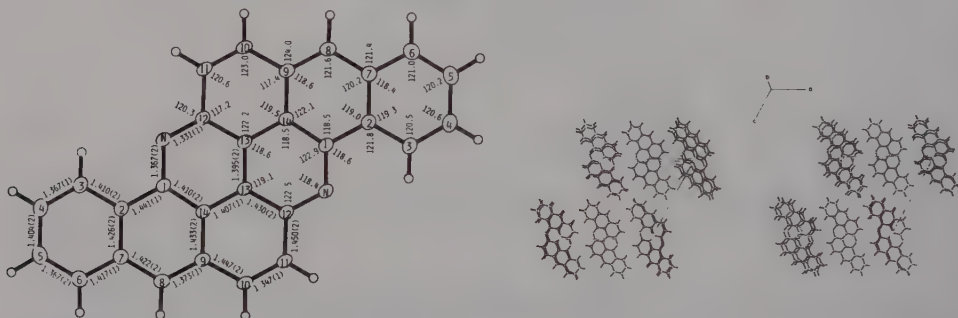


Fig. 1.  $C_{28}H_{14}N_2$ : bond distances and angles in the molecule (left) and a stereoscopic view of the crystal packing viewed along the  $b$  axis (right).

### 1,4-DIPHENYL-1,4-DIHYDROBENZO[g]PYRIDAZINO[1,2-b]PHTHALAZINE-6,13-DIONE

$C_{28}H_{20}N_2O_2$  (I)

### 1,4-DIHYDROBENZO[g]PYRIDAZINO[1,2-b]PHTHALAZINE-6,13-DIONE

$C_{16}H_{12}N_2O_2$  (II)

M.C. APREDA, C. FOCES-FOCES, F.H. CANO and S. GARCÍA-BLANCO, 1982. *Acta Cryst.*, B38, 2834-2838.

I. Monoclinic,  $C2/c$ ,  $a = 20.2629$ ,  $b = 7.3456$ ,  $c = 28.9414$  Å,  $\beta = 106.77^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.044$  for 2738 reflexions.

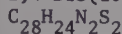
II. Orthorhombic,  $P2_12_12_1$ ,  $a = 24.2169$ ,  $b = 12.5360$ ,  $c = 3.9942$  Å,  $Z = 4$ . Cu radiation,  $R = 0.039$  for 1092 reflexions.

Molecules I and II (Fig. 1) have similar molecular geometry and bond lengths and angles are generally as expected. The pyridazine rings have different conformations and in II the diazaquinonic ring is more planar than in I. The average angle between the phenyl planes is  $52.7^\circ$ .



Fig. 1. Molecular structures of  $C_{28}H_{20}N_2O_2$  (I) and  $C_{16}H_{12}N_2O_2$  (II).

## 1,4-BIS(10-PHENOTHAZINYL)BUTANE



B. BEAGLEY, C.H. FRYDRYCH, R.G. PRITCHARD and D.R. TAYLOR, 1982. *Acta Cryst.*, **B38**, 2925-2927.

Monoclinic,  $P2_1/c$ ,  $a = 10.807$ ,  $b = 20.176$ ,  $c = 12.222 \text{ \AA}$ ,  $\beta = 123.31^\circ$ ,  $D_m = 1.36$ ,  $Z = 4$ . Mo radiation,  $R = 0.070$  for 917 reflexions.

In the crystal structure two crystallographically different centrosymmetric molecules A and B occur (Fig. 1). Columns of alternating A and B molecules paired in the form of a cross (fourfold pseudo screw operation) lie parallel to  $a$ . The aliphatic chains lie inside the columns, isolated from the chains of adjacent columns by the phenothiazine groups.

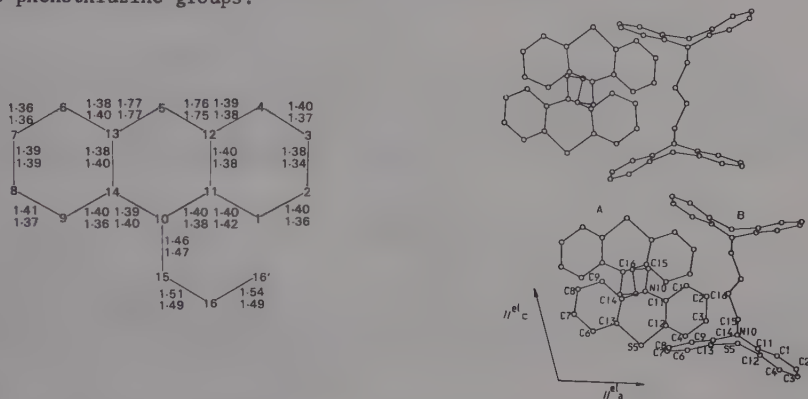
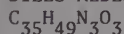


Fig. 1.  $\text{C}_{28}\text{H}_{24}\text{N}_2\text{S}_2$ : bond lengths for molecules A (upper entries) and B and the projection of the crystal structure on (010).

DIELS-ALDER ADDUCT OF 4-PHENYL-1,2,4-TRIAZOLINE-3,5-DIONE WITH PREVITAMIN  $\text{D}_3$ 

M. VAN MEERSSCHE, B. TINANT, G. GERMAIN, J.P. DECLERCQ, L.J. VANMAELE, P.J. DE CLERCQ and M.E. VANDEWALLE, 1982. *Bull. Soc. Chim. Belg.*, **91**, 205-211.

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.142$ ,  $b = 11.873$ ,  $c = 37.852 \text{ \AA}$ ,  $Z = 4$ . Mo radiation,  $R = 0.068$  for 1920 reflexions.

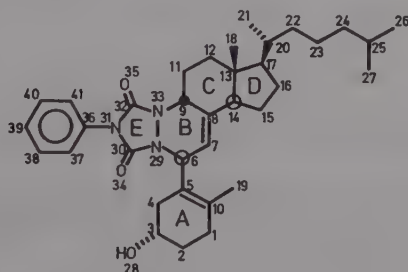


Fig. 1. The  $\text{C}_{35}\text{H}_{49}\text{N}_3\text{O}_3$  molecule.

The absolute stereochemistry of the title compound is shown in Fig. 1; the configuration at the asymmetric carbons is C(20)R, C(17)R, C(13)R, C(14)S, C(9)R, C(6)S, and C(3)S. The cyclohexene ring A



(boat) form. The cyclopentane ring D adopts a conformation intermediate between a half-chair ( $C_2$  symmetry) and an envelope ( $C_s$  symmetry) and the urazole ring E is almost flat.

3,7,11-TRIS(p-CHLOROPHENYL)-2,3,6,7,10,11-HEXAHYDRO-2,2,6,6,10,10-HEXAPHENYLTRIS-[1,2,4]TRIAZOLO[1,5-a,1',5'-c:1'',5'''-e][1,3,5]TRIAZINE

$C_{60}H_{42}Cl_3N_9$

J.L. FLIPPEN-ANDERSON, J.H. KONNERT and R. GILARDI, 1982. *Acta Cryst.*, **B38**, 2865-2869.

Triclinic,  $P\bar{1}$ ,  $a = 13.242$ ,  $b = 13.558$ ,  $c = 16.103$  Å,  $\alpha = 108.5$ ,  $\beta = 91.2$ ,  $\gamma = 111.6^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.105$  for 6053 reflexions.

The molecule (Fig. 1) is asymmetric with two of the  $-C_6H_4Cl$  groups on one side of the central ring system and the third on the opposite side. Bond lengths and angles are generally as expected though there are three notably short C=N bonds (1.261(5), 1.274(5) and 1.265(5) Å) radiating from the central ring; "normal" C=N bonds are in the range 1.29-1.31 Å. Packing is influenced only by van der Waals forces.

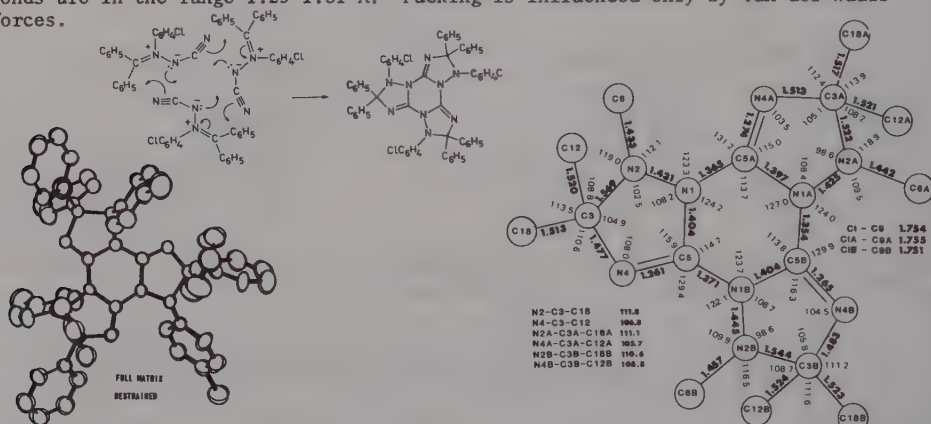


Fig. 1.  $C_{60}H_{42}Cl_3N_9$ : conformation of the molecule and bond lengths and angles in the central heterocyclic ring system of the molecule.

1-METHYL-1,3,5,7-TETRAAZAADAMANTAN-1-IUM TRIIODIDE

$C_7H_{15}I_3N$

$[(CH_2)_6N_4CH_3]I_3$

P.K. HON, T.C.W. MAK and J. TROTTER, 1982. *Z. Krist.*, **158**, 213-220.

Monoclinic,  $P2_1/c$ ,  $a = 12.989$ ,  $b = 12.735$ ,  $c = 8.590$  Å,  $\beta = 94.73^\circ$ ,  $D_m = 2.39$ ,  $Z = 4$ . Mo radiation,  $R = 0.054$  for 2183 reflexions.

The C-N bonds involving the formally positive quaternary N atom (Fig. 1) are longer than the other C-N bonds (1.50 to 1.53(1) Å compared with 1.42 to 1.48(1) Å). The  $I_3^-$  ions occupy two non-equivalent sites of symmetry  $\bar{1}$  (with I-I 2.910 and 2.926(2) Å) and may be slightly disordered.

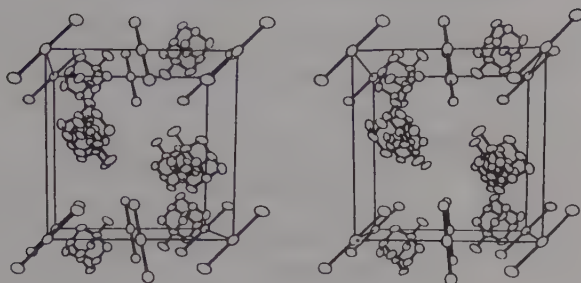


Fig. 1. A stereoview of the  $C_7H_{15}I_3N$  structure.

I.L. KARLE, 1982. Acta Cryst., B38, 1022-1024.

Triclinic,  $P\bar{1}$ ,  $a = 6.953$ ,  $b = 7.811$ ,  $c = 8.328$  Å,  $\alpha = 94.34$ ,  $\beta = 91.43$ ,  $\gamma = 115.29^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.064$  for 1039 reflexions.

In the molecule (Fig. 1) the five-membered ring containing a planar C(1) atom and a planar N(2) atom is approximately planar. The five-membered ring containing all saturated C atoms has the usual envelope conformation with C(8), C(4), C(5) and C(6) nearly in a plane. The molecules are linked in the crystal by O-H...O and N-H...O hydrogen bonds with O...O 2.751 Å and N...O 2.813 Å.

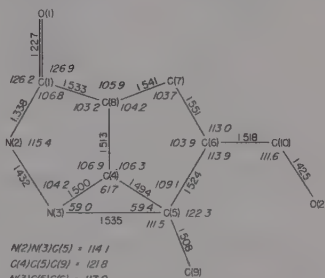
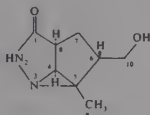


Fig. 1. Bond lengths and angles in the  $C_8H_{12}N_2O_2$  molecule ( $\sigma = 0.006 \text{ \AA}$  and  $0.3^\circ$ ).

J. VILCHES, F. FLORENCIO, P. SMITH-VERDIER and S. GARCIA-BLANCO, 1982. *Cryst. Struct. Comm.*, 11, 13-17.

Orthorhombic, Pnma,  $a = 9.901$ ,  $b = 8.362$ ,  $c = 12.059$  Å,  $Z = 4$ . Cu radiation,  $R = 0.100$ , for 752 reflexions.

The molecule lies about a crystallographic mirror plane (Fig. 1). The piperidine ring has a distorted chair conformation and the five-membered ring of the bicyclo system has an envelope form with N(6) 0.635 Å from the plane of the other four atoms.

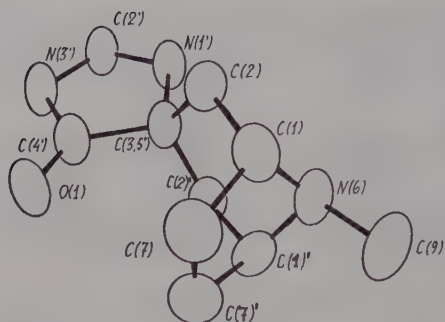


Fig. 1. A view of  $C_{10}H_{15}N_3O$ .

8-HYDROXY-1,4,5,7-TETRAMETHYL-6,8-DIAZABICYCLO[3.2.1]OCT-6-ENE 6-OXIDE  
 $C_{10}H_{18}N_2O_2$

T. OTA, S. MASUDA, H. TANAKA and M. KIDO, 1982. Bull. Chem. Soc. Jpn., 55, 171-173.

Monoclinic,  $P2_1/n$ ,  $a = 9.459$ ,  $b = 10.335$ ,  $c = 11.352$  Å,  $\beta = 105.50^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.083$  for 710 reflexions.

The seven-, six- and five-membered rings (Fig. 1) have boat, chair and half-chair conformations respectively, with  $N(8)-O(13)$  1.455(6),  $N(6)-O(14)$  1.293(6) Å.

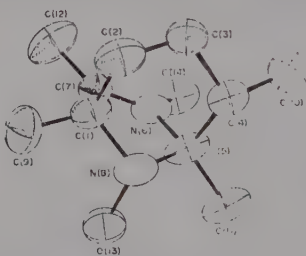


Fig. 1. A perspective drawing of  $C_{10}H_{18}N_2O_2$ .

4-METHYL-2,4,6-TRIAZAPENTACYCLO[5.3.2.1<sup>9,11</sup>.0<sup>2,6</sup>.0<sup>8,10</sup>]TRIDECANE-3,5-DIONE  
 $C_{11}H_{13}N_3O_2$

W. ADAM, O. De LUCCHI, K. PETERS, E.-M. PETERS and H.G. von SCHNERING, 1982. J. Am. Chem. Soc., 104, 161-166.

Triclinic,  $P\bar{1}$ ,  $a = 6.791$ ,  $b = 12.411$ ,  $c = 6.320$  Å,  $\alpha = 107.53$ ,  $\beta = 95.30$ ,  $\gamma = 98.82^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.057$  for 1966 reflexions.

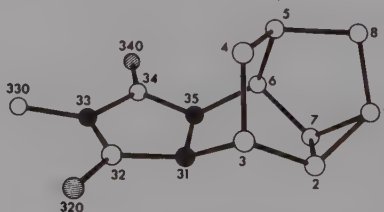


Fig. 1. The  $C_{11}H_{13}N_3O_2$  molecule.

The molecule is shown in Fig. 1. Bond lengths in the cyclopropane ring are: C(1)-C(2) 1.520(4), C(1)-C(7) 1.486(4) and C(2)-C(7) 1.497(3) Å. Remaining bond lengths ( $\sigma = 0.002$ -0.004 Å) and angles ( $\sigma = 0.2^\circ$ ) are normal.

1-AZONIATRICYCLO[4.4.4.0<sup>1,6</sup>]TETRADECANE BROMIDE  
C<sub>13</sub>H<sub>24</sub>BrN (I)

1-AZONIATRICYCLO[4.4.3.0<sup>1,6</sup>]TRIDECAENE BROMIDE  
C<sub>12</sub>H<sub>22</sub>BrN (II)

J.M. McINTOSH, M.A. KHAN and L.T.J. DELBAERE, 1982. *Canad. J. Chem.*, **60**, 1073-1077.

I. Hexagonal, P6<sub>3</sub>/mmc,  $a = 8.383$ ,  $c = 10.092$  Å,  $Z = 2$ . Cu radiation,  $R = 0.068$  for 239 reflexions.

II. Hexagonal, P6<sub>3</sub>/mmc,  $a = 8.283$ ,  $c = 10.046$  Å,  $Z = 2$ . Mo radiation,  $R = 0.058$  for 244 reflexions.

Both compounds (Fig. 1) are statistically disordered with both enantiomers occurring in the unit cell. I has C<sub>3</sub> symmetry and exists as two enantiomers which are interconverted by chair-chair ring inversion of all three six-membered rings. In II the five-membered ring and the two six-membered rings are mutually scrambled.

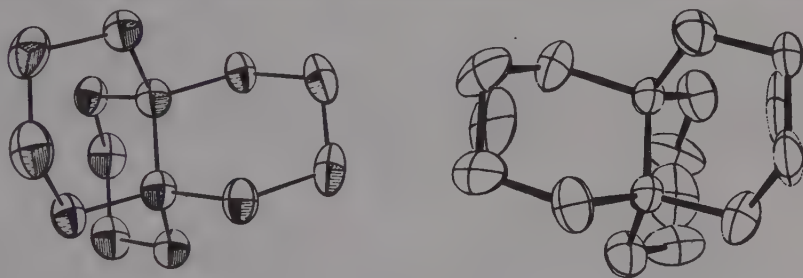


Fig. 1. Views of the cations in C<sub>13</sub>H<sub>24</sub>BrN (left) and C<sub>12</sub>H<sub>22</sub>BrN (right).

4-PHENYL-2,4,6-TRIAZATRICYCLO[5.3.2.0<sup>2,6</sup>]DODECA-9,11-DIEN-3,5,8-TRIONE  
C<sub>15</sub>H<sub>11</sub>N<sub>3</sub>O<sub>3</sub>

K. PETERS, E.-M. PETERS, H.G. VON SCHNERING, W. ADAM and H. REBOLLO, 1982. *Z. Krist.*, **160**, 149-152.

Triclinic, P $\bar{1}$ ,  $a = 7.126$ ,  $b = 14.178$ ,  $c = 6.876$  Å,  $\alpha = 99.98^\circ$ ,  $\beta = 107.69^\circ$ ,  $\gamma = 92.70^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.080$  for three-dimensional data.

The analysis establishes that the molecule has anti geometry shown in Fig. 1. The four structural fragments A (1, 11, 12, 7), B (1, 10, 9, 8, 7), C (1, 2, 6, 7) and D (2, 3, 4, 5, 6) are all planar within standard deviations. The dihedral angles were calculated to be A/B 62.3, A/C 51.7, B/C 66.0 and C/D 31.8°. The plane of the phenyl ring is orientated at an angle of 43.9° to the plane of the triazolinodione ring.

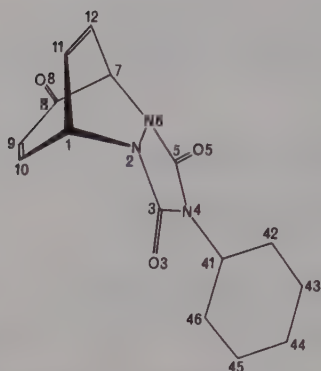


Fig. 1. A perspective drawing of  $C_{15}H_{11}N_3O_3$ .

syn-1,6-IMINO-8,13-METHANO[14]ANNULENE  
 $C_{15}H_{13}N$

R. DESTRO, A. GAVEZZOTTI and M. SIMONETTA, 1982. *Acta Cryst.*, **B38**, 1352-1354.

Monoclinic,  $C2/c$ ,  $a = 19.125$ ,  $b = 6.096$ ,  $c = 19.151$  Å,  $\beta = 102.98^\circ$ ,  $D_m = 1.23$ ,  $Z = 8$ .  
 Mo radiation,  $R = 0.065$  for 1352 reflexions.

The molecule (Fig. 1) is subject to the strain imposed by the syn bridging groups but nonetheless can be regarded as "aromatic". The molecules are held together in the crystal by van der Waals interactions.

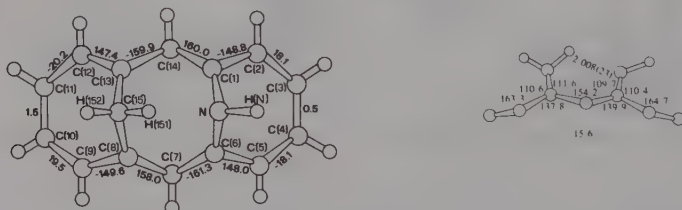


Fig. 1.  $C_{15}H_{13}N$ : torsion angles (left) and dihedral angles between least-squares planes, with a short  $N \cdots H$  content (right).

4-PHENYL-2,4,6-TRIAZATETRACYCLO[5.3.2.0<sup>2,6</sup>.0<sup>8,10</sup>]DODEC-11-ENE-3,5-DIONE  
 $C_{15}H_{13}N_3O_2$

K. PETERS, E.-M. PETERS, H.G. VON SCHNERING, W. ADAM and H. REBOLLO, 1982. *Z. Krist.*, **160**, 145-148.

Orthorhombic,  $Pbca$ ,  $a = 14.869$ ,  $b = 15.273$ ,  $c = 10.855$  Å,  $Z = 8$ . Mo radiation,  $R = 0.052$  for three-dimensional data.

The analysis establishes the syn-conformation for the molecule (Fig. 1). Beside plane A (8,9,10), the structural fragments B(1,10,8,7), C(1,11,12,7), D(1,2,6,7) and E(2,3,4,5,6) are all planar within standard deviations. The dihedral angles were calculated to be A/B 65.2, B/C 54.0, B/D 65.8, C/D 60.2 and D/E 45.3°. The plane of the phenyl ring is orientated at an angle of 45.1° to the plane of the triazolinedione ring.

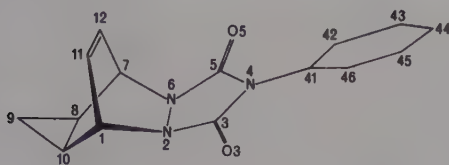


Fig. 1. A perspective drawing of  $C_{15}H_{13}N_3O_2$ .

(-) -N-METHYL-3,4-METHYLENEDIOXYMORPHINAN-6-ONE  
 $C_{18}H_{21}NO_3$  (I)

(-) -N-METHYL-4-METHOXYMORPHINAN-6-ONE  
 $C_{18}H_{23}NO_2$  (II)

A. BROSSI, L. ATWELL, A.E. JACOBSON, M.D. ROZWADOWSKA, H. SCHMIDHAMMER, J.L. FLIPPEN-ANDERSON and R. GILARDI, 1982. *Helv. Chim. Acta*, **65**, 2394-2404.

I. Orthorhombic,  $P2_12_12_1$ ,  $a = 7.062$ ,  $b = 13.722$ ,  $c = 15.269$  Å,  $Z = 4$ . Cu radiation,  $R = 0.083$  for 1149 reflexions.

II. Orthorhombic,  $P2_12_12_1$ ,  $a = 7.076$ ,  $b = 13.463$ ,  $c = 15.780$  Å,  $Z = 4$ . Cu radiation,  $R = 0.064$  for 1162 reflexions.

The analyses showed that both the 3,4-methylenedioxy group in I and the methoxy group in II are nearly coplanar with their aromatic ring (Fig. 1). Both molecules are T-shaped with the six-membered ring bearing the carbonyl O atom in chair form.

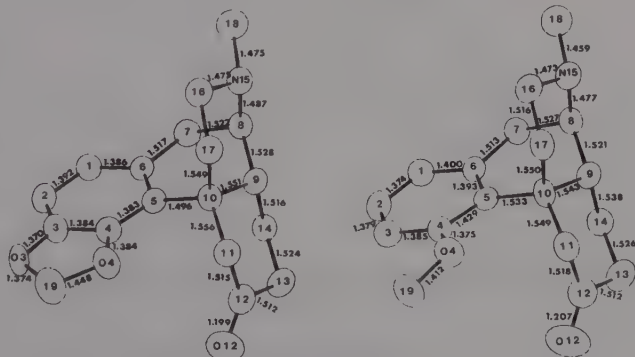


Fig. 1. The  $C_{18}H_{21}NO_3$  (I) (left) and  $C_{18}H_{23}NO_2$  (II) (right) molecules and bond lengths.

1,3,3,5,5-PENTAMETHYL-2-exo-METHOXY-4-PHENYL-7-AZABICYCLO[2.2.1]HEPTANE HYDROGEN PERCHLORATE  
 $C_{18}H_{28}ClNO_5$

R. SWANSON, J.L. STAVINOKA and P.S. MARIANO, 1982. *Cryst. Struct. Comm.*, **11**, 799-804.

Monoclinic,  $P2_1/c$ ,  $a = 10.488$ ,  $b = 12.275$ ,  $c = 16.106$  Å,  $\beta = 111.79^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.132$  for 1794 reflexions.

The R-factor is high because of disorder associated with the  $ClO_4^-$  anion. Molecular dimensions are unexceptional. A view of the structure is in Fig. 1.



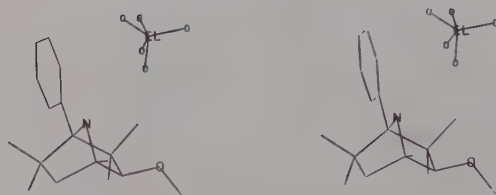


Fig. 1. A stereoview of  $C_{18}H_{28}ClNO_5$ ; the perchlorate moiety is disordered.

9,10,12,13-DIBENZO-4-METHYL-3,5-DIOXO-2,4,6-TRIAZATETRACYCLO[6.5.0.0<sup>2,6</sup>.0<sup>7,11</sup>]-TRIDECAHE

$C_{19}H_{15}N_3O_2$

W. ADAM, O. DE LUCCHI, K. PETERS, E.-M. PETERS and H.G. von SCHNERING, 1982. J. Am. Chem. Soc., 104, 5747-5753.

Orthorhombic,  $Pbca$ ,  $a = 14.992$ ,  $b = 23.974$ ,  $c = 8.479$  Å,  $Z = 8$ . Mo radiation,  $R = 0.067$  for 2307 reflexions.

The molecule is shown in Fig. 1. Bond lengths and angles ( $\sigma$  0.004-0.008 Å and 0.2-0.8°) are as expected.

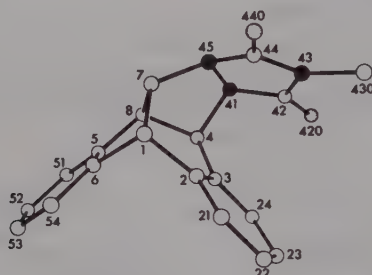


Fig. 1. The  $C_{19}H_{15}N_3O_2$  molecule.

6,9-DIMETHOXY-2,13-DIAZA[4]PARACYCLO[4](3,5)PYRIDINOPHANE-1,14-DIONE  
 $C_{19}H_{21}N_3O_4$  (I)

19-BENZYL-16,19-DIHYDRO-2,13-DIAZA[4]PARACYCLO[4](3,5)PYRIDINOPHANE-1,4-DIONE  
 $C_{24}H_{25}N_3O_2$  (II)

A.M. VAN HERK, K. GOUBITZ, A.R. OVERBEEK and C.H. STAM, 1982. Acta. Cryst., B38, 490-494.

I. Monoclinic,  $P2_1/n$ ,  $a = 10.2801$ ,  $b = 9.4333$ ,  $c = 18.7174$  Å,  $\beta = 101.42^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.041$  for 2519 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 12.980$ ,  $b = 5.719$ ,  $c = 26.721$  Å,  $\beta = 99.26^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.048$  for 2704 reflexions.

In I (Fig. 1), the pyridine and benzene rings are inclined at  $77^\circ$  with  $H(4) \dots C(\text{benzene})$  2.80-3.11 Å. The molecules are linked by  $N(8) \dots O(21)$  and  $N(19) \dots O(22)$  hydrogen bonds of length 2.993 and 2.938 Å to form sheets parallel to (001). In II the 1,4-dihydropyridine ring is in the boat conformation. One of its four atoms is almost above the centre of the benzene ring with  $H \dots C(\text{benzene})$  in the range 2.94-3.18 Å. The conformations of the two ring-connecting chains are very similar in I but different in II. Molecular geometry is normal.

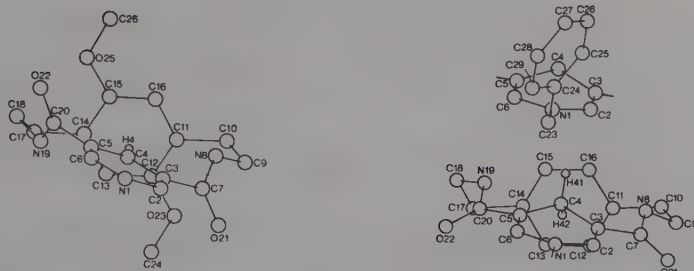


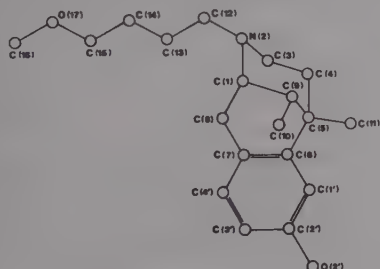
Fig. 1. Numbering of atoms and geometry of  $C_{19}H_{21}N_3O_4$  (I) (left) and  $C_{24}H_{25}N_3O_2$  (II) (right).

(-)-2'-HYDROXY-2-(4-METHOXYBUTYL)-5,9-DIMETHYL-6,7-BENZOMORPHAN HYDROBROMIDE  
 $C_{19}H_{30}BrNO_2$

O.M. PEETERS, C.J. DE RANTER and N.M. BLATON, 1982. Acta Cryst., B38, 3168-3171.

Monoclinic,  $P2_1$ ,  $a = 12.2567$ ,  $b = 8.9898$ ,  $c = 17.5559$  Å,  $\beta = 92.63^\circ$ ,  $D_m = 1.32$ ,  $Z = 4$ . Cu radiation,  $R = 0.041$  for 3035 reflexions.

There are two independent molecules (Fig. 1) in the asymmetric unit and they have different N side-chain conformations. For molecule A C1-N2-C12-C13 and N2-C12-C13-C14 are both antiplanar, whereas in molecule B, the former is synclinal and the latter antiplanar. Both conformations correspond to minimum energy orientations from theoretical calculations.



	Molecule A	Molecule B
C(1)-N(2)-C(12)-C(13)	-157.9 (5)	-55.0 (7)
C(3)-N(2)-C(12)-C(13)	73.9 (6)	177.1 (5)
N(2)-C(12)-C(13)-C(14)	176.6 (5)	-168.1 (6)
C(12)-C(13)-C(14)-C(15)	-174.5 (6)	-160.6 (10)
C(13)-C(14)-C(15)-O(17)	-178.7 (7)	-22.1 (20)
C(14)-C(15)-O(17)-C(16)	-174.2 (8)	141.5 (15)

Fig. 1. The numbering scheme of the benzomorphane derivative  $C_{19}H_{30}BrNO_2$  and N side-chain torsion angles.

2-BENZYL-3-OXO-2-AZABICYCLO[2.2.1]HEPT-6-*exo*-YL p-TOLUENESULFONATE  
 $C_{20}H_{21}NO_4S$  (I)

2-BENZYL-6-*exo*-PHENOXY-2-AZABICYCLO[2.2.1]HEPTAN-3-ONE  
 $C_{19}H_{19}NO_2$  (II)

2-BENZYL-6-*exo*-(2-iodo-4-methylphenoxy)-2-AZABICYCLO[2.2.1]HEPTAN-3-ONE  
 $C_{20}H_{20}INO_2$  (III)

2-BENZYL-6-*exo*-(2,6-diiodo-4-methylphenoxy)-2-AZABICYCLO[2.2.1]HEPTAN-3-ONE  
 $C_{20}H_{19}I_2NO_2$  (IV)

2-BENZYL-6-*exo*-PHENOXY-2-AZABICYCLO[2.2.2]OCTAN-3-ONE  
 $C_{20}H_{21}NO_2$  (V)

H.L. AMMON, P.H. MAZZOCCHI, L. LIU, E.C. COLICELLI, R. DOHERTY and J.M. STEWART, 1982. Acta Cryst., B38, 540-547.

Fig. 1. Views of the 2-azabicyclo[2.2.1]heptane derivatives I to IV and of the 2-azabicyclo[2.2.2]octane derivative (V).

## 10,11-DIHYDRO-5-(3-QUINUCLIDINYL)-5H-DIBENZO[b,f]AZEPINE (QUINUPRAMINE)

 $C_{21}H_{24}N_2$ 

J.P. REBOUL, J.C. SOYFER, B. CRISTAU, C. CARANONI and G. PÈPE, 1982. Acta Cryst., B38, 2633-2638.

Monoclinic,  $P2_1/c$ ,  $a = 12.990$ ,  $b = 15.808$ ,  $c = 8.003$  Å,  $\beta = 91.45^\circ$ ,  $D_m = 1.23$ ,  $Z = 4$ . Cu radiation,  $R = 0.031$  for 1868 reflexions.

In the molecule (Fig. 1) the dihedral angle between the benzene rings is  $114.5(1)^\circ$ . The azepine ring has a gauche boat conformation with C(5), C(6) and N(15) on the same side of the plane defined by atoms C(11) to C(14). Conformational parameters are compared to those of similar tricyclic anti-depressant drugs.

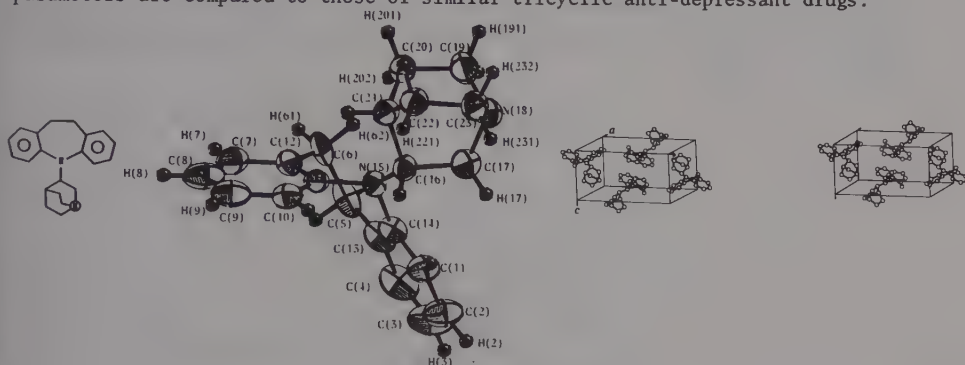


Fig. 1.  $C_{21}H_{24}N_2$ : a perspective view of the molecule and a stereoscopic view of the crystal structure.

## 3,7-DIMETHYL-1,5-DIPHENYL-3,7-DIAZABICYCLO[3,3,1]NONAN-9-ONE

 $C_{21}H_{24}N_2O$ 

O.I. LEVINA, K.A. POTEKHIN, V.G. RAU, Yu.T. STRUCHKOV, V.A. PALLYULIN and N.S. ZEFIROV, 1982. Cryst. Struct. Comm., 11, 1073-1076.

Monoclinic,  $P2_1/c$ ,  $a = 7.9037$ ,  $b = 10.8203$ ,  $c = 20.905$  Å,  $\beta = 97.11^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.055$  for 1807 reflexions.

The molecule (Fig. 1) has a chair-boat conformation. The ring C9-C1-C2-N3-C4-C5 is nearly an ideal chair:  $S$  1.250,  $\theta$   $0.7^\circ$ ,  $\psi_2$   $1.1^\circ$ ; the ring C9-C5-C6-N7-C8-C1 has a slightly distorted boat form with more significant pucker of N7 apex,  $S$  0.963,  $\theta$   $93.3^\circ$ ,  $\psi_2$   $271.1^\circ$ .

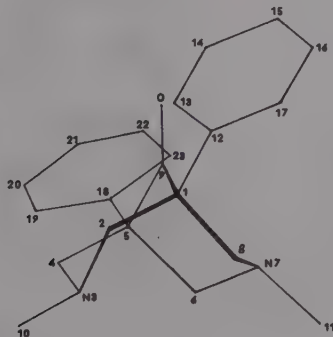


Fig. 1. A view of  $C_{21}H_{24}N_2O$ .

3,7-DIMETHYL-1,5-DIPHENYL-3-AZA-7-AZONIABICYCLO[3.3.1]NONAN-9-ONE HYDROGEN SULPHATE  
HEMIHYDRATE

$C_{21}H_{26}N_2O_5S \cdot 0.5(H_2O)$

$[C_{21}H_{25}N_2O]^+ HSO_4^-, 0.5(H_2O)$

O.I. LEVINA, E.N. KURKUTOVA, K.A. POTEKHIN, Yu.T. STRUCHKOV, V.A. PALLYULIN and N.S. ZEFIROV, 1982. Cryst. Struct. Comm., 11, 1915-1919.

Monoclinic,  $P2_1/c$ ,  $a = 10.6846$ ,  $b = 12.3235$ ,  $c = 16.527$  Å,  $\beta = 105.915^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.053$  for 2105 reflexions.

The molecule (Fig. 1) has a double chair conformation with  $N3...N7$  2.67 Å. The ring  $C9-C1-C8-N7-C6-C5$  is nearly an ideal chair ( $S = 1.115$ ,  $\theta = 0.8^\circ$ ,  $\psi_2 = 243.9^\circ$ ); the ring  $C9-C5-C4-N3-C2-C1$  has a chair form with C9 as the most puckered apex ( $S = 1.244$ ,  $\theta = 1.3^\circ$ ,  $\psi_2 = 275.2^\circ$ ). The hydrogen atom of the  $HSO_4^-$  ion is disordered occupying two positions at O2 and O4 atoms and the position of the water molecule is statistically half-occupied.

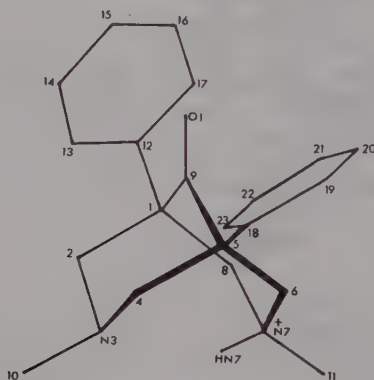


Fig. 1. A view of the  $[C_{21}H_{25}N_2O]^+$  ion.

5a,5b,6,8a,9,9a,9b,9c-OCTAHYDRO-2-PHENYL-6,9-ETHENO-2H-CYCLOPROPA[3',4']CYCLOHEPTA-[1',2':3,4]CYCLOBUT[1,2-e]INDAZOLE

$C_{23}H_{20}N_2$

K.M. BEEM and B. RUBIN, 1982. Cryst. Struct. Comm., 11, 607-612.

Rhombohedral  $R\bar{3}$ ,  $a = 20.927$ ,  $c = 118.68$  Å,  $Z = 6$ . Cu radiation,  $R = 0.107$  for 1883 reflexions.

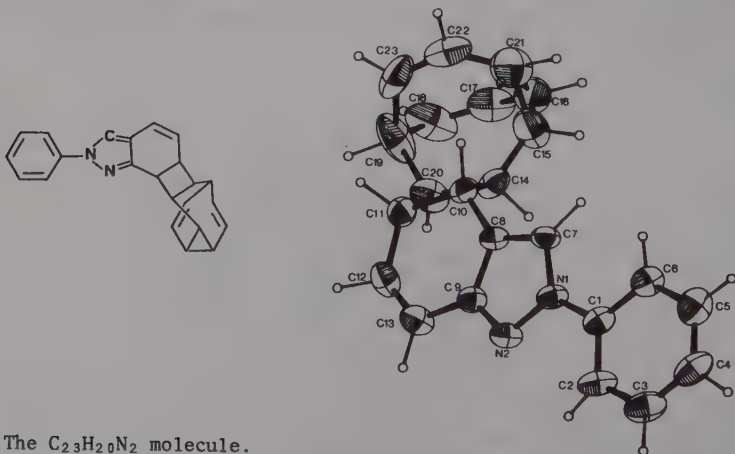


Fig. 1. The  $C_{23}H_{20}N_2$  molecule.

The molecule (Fig. 1) consists of a planar 2-phenyl-2H-indazole moiety added across one of the double bonds of a bullvalene tautomer. The lone pair of electrons of N(1) is appreciably conjugated into the ring.

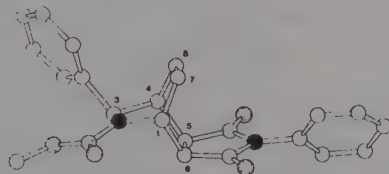
2-(CARBOMETHOXY)-3-endo-PHENYL-2-AZABICYCLO[2.2.2]OCT-7-ENE-5,6-DICARBOXYLIC ACID  
N-PHENYLIMIDE

$C_{23}H_{20}N_2O_4$

G.R. KROW, J.T. CAREY, D.E. ZACHARIAS and E.E. KNAUS, 1982. J. Org. Chem., 47, 1989-1993.

Monoclinic,  $P2_1/n$ ,  $a = 17.920$ ,  $b = 6.177$ ,  $c = 17.915$  Å,  $\beta = 90.48^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.149$  for 1770 reflexions.

The analysis establishes the structure shown in Fig. 1, with a planar s-cis conformation for the N-carbomethoxy function.





## 3,7-DIBENZYL-1,5-DIPHENYL-3,7-DIAZABICYCLO[3.3.1]NONAN-9-ONE

 $C_{33}H_{32}N_2O$ 

O.I. LEVINA, K.A. POTEKHIN, E.N. KURKUTOVA, Yu.T. STRUCHKOV, V.A. PALYULIN and N.S. ZEFIROV, 1982. Cryst. Struct. Comm., 11, 1909-1913.

Orthorhombic,  $P2_12_12_1$ ,  $a = 10.529$ ,  $b = 24.099$ ,  $c = 10.161$  Å,  $Z = 4$ . Cu radiation,  $R = 0.045$  for 1590 reflexions.

The ring C9-C5-C4-N3-C2-C1 (Fig. 1) is a slightly distorted chair ( $S = 1.255$ ,  $\theta = 1.4^\circ$ ,  $\psi_2 = 146.7^\circ$ ). The ring C9-C1-C8-N7-C6-C5 has a slightly distorted boat form with the greatest puckering for the N7 apex, ( $S = 0.982$ ,  $\theta = 93.7^\circ$ ,  $\psi_2 = 268.2^\circ$ ).

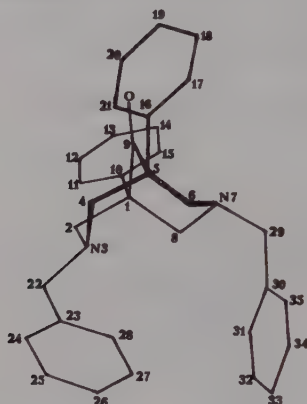


Fig. 1. A view of  $C_{33}H_{32}N_2O$ .

(5R)-(2(1S,2S,4R),5 $\alpha$ ,5 $\alpha\beta$ ,7 $\alpha\beta$ ,8 $\alpha$ )-5,5a,7a,8-TETRAHYDRO-5,7-DIMETHYL-5a,6-DIPHENYL-2-(1,7,7-TRIMETHYLBICYCLO(2.2.1)HEPT-2-YL)-5,8-ETHENO-1H-CYCLOBUTA(d)(1,2,4)TRIAZOLO-(1,2-a)PYRIDAZINE-1,3(2H)-DIONE

 $C_{34}H_{37}N_3O_2$ 

L.A. PAQUETTE, Y. HANZAWA, G.J. HEFFERON and J.F. BLOUNT, 1982. J. Org. Chem., 47, 265-272.

Orthorhombic,  $P2_12_12_1$ ,  $a = 12.814$ ,  $b = 14.516$ ,  $c = 15.337$  Å,  $Z = 4$ . Cu radiation,  $R = 0.041$  for 2034 reflexions.

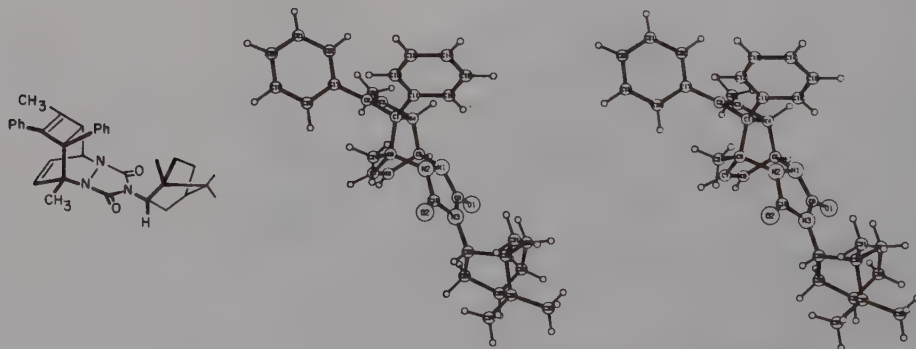
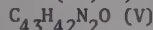
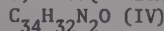
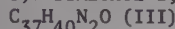


Fig. 1. Formula and stereoview of  $C_{34}H_{37}N_3O_2$ .

rel-1-(2S,4R,6R,8S)-2,4,6,8-TETRAKIS(3,5-DIMETHYLPHENYL)-3,7-DIAZABICYCLO[3.3.1]NONAN-9-ONE



1. Triclinic,  $P\bar{1}$ ,  $a = 14.731$ ,  $b = 15.169$ ,  $c = 8.258$  Å,  $\alpha = 104.59$ ,  $\beta = 105.88$ ,  $\gamma = 102.35^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.075$  for 5619 reflexions.

II. Monoclinic,  $P2_1/m$ ,  $a = 19.876$ ,  $b = 16.198$ ,  $c = 9.658$  Å,  $\beta = 100.55^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.117$  for 4071 reflexions.

III. Orthorhombic,  $Pnma$ ,  $a = 6.719$ ,  $b = 29.616$ ,  $c = 14.650$  Å,  $Z = 4$ . Mo radiation,  $R = 0.090$  for 2352 reflexions.

IV. Triclinic,  $P\bar{1}$ ,  $a = 13.191$ ,  $b = 18.633$ ,  $c = 12.305$  Å,  $\alpha = 92.46$ ,  $\beta = 114.37$ ,  $\gamma = 96.96^\circ$ .  $Z = 4$ . Mo radiation,  $R = 0.088$  for 4754 reflexions.

V. Triclinic,  $P\bar{1}$ ,  $a = 13.194$ ,  $b = 13.613$ ,  $c = 10.992$  Å,  $\alpha = 96.64$ ,  $\beta = 114.78$ ,  $\gamma = 104.95^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.088$  for 2931 reflexions.

Each tetraaryl-3,7-diazabicyclononane exists in the chair-boat conformation with equatorial aryl groups. Two of the five aryl substituents in V (Fig. 1) occupy a 1,3-diaxial position while the three neighbouring aryl groups exhibit an unexpected propeller-like arrangement.

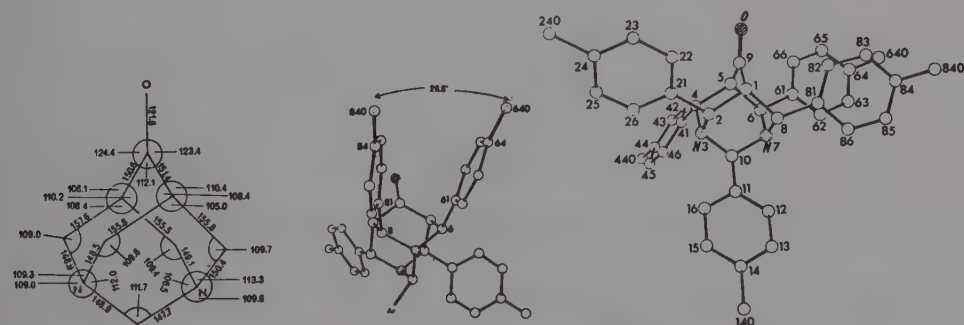
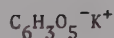


Fig. 1. A perspective view, principal bond lengths (pm), angles ( $^{\circ}$ ) and other molecular dimensions in  $C_4H_4N_2O$ .

## POTASSIUM HYDROGEN FURAN-2,5-DICARBOXYLATE



S. JAULMES, G. CASSANAS and P. LARUELLE, 1982. *Acta Cryst.*, B38, 279-281.

Monoclinic, B2/b,  $a = 9.759$ ,  $b = 6.6952$ ,  $c = 11.193 \text{ \AA}$ ,  $\gamma = 114.49^\circ$ ,  $D_m = 1.95$ ,  $Z = 4$ . Mo radiation,  $R = 0.0486$  for 1057 reflexions.

In the crystal structure (Fig. 1) the potassium ion is coordinated by seven oxygen atoms.  $\text{C}_6\text{H}_3\text{O}_5^-$  anions lying along twofold axes are linked into infinite chains by hydrogen bonds with  $\text{O} \cdots \text{O} \ 2.459 \text{ \AA}$ . The two carboxyl groups are slightly twisted out of the plane of the furan ring.

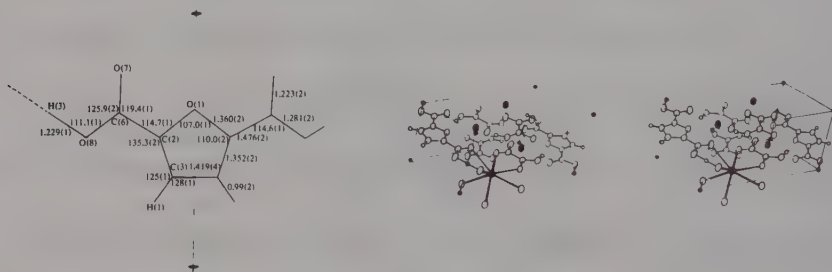


Fig. 1. Bond lengths and angles in the  $\text{C}_6\text{H}_3\text{O}_5^-$  ion and a stereoscopic view of the crystal structure.

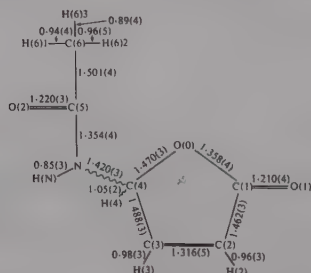
## 4-ACETAMIDO-2-BUTEN-4-OLIDE



Z. RUZIC-TOROS and B. KOJIC-PRODIC, 1982. *Acta Cryst.*, B38, 1664-1666.

Monoclinic,  $P2_1/c$ ,  $a = 8.790$ ,  $b = 5.314$ ,  $c = 14.264 \text{ \AA}$ ,  $\beta = 96.47^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.039$  for 963 reflexions.

The structure contains both the D and L enantiomers. The lactone ring is planar with a mean ring torsion angle of  $0.7(3)^\circ$ . The molecular geometry (Fig. 1) is normal. Molecules are linked by  $\text{N-H} \cdots \text{O} \ (3.000(4) \text{ \AA})$  hydrogen bonds into infinite chains parallel to  $b$ .



Monoclinic,  $P2_1/c$ ,  $a = 8.050$ ,  $b = 9.086$ ,  $c = 9.713$  Å,  $\beta = 102.86^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.046$  for 872 reflexions.

The analysis shows that this compound is the racemate of D-,D- and L-,L- lactide and not the meso form. Both D-,D- and L-,L- type molecules are present in the crystal. The molecule (Fig. 1) has  $C_2$  symmetry within experimental error and the cyclic group has an irregular, skew-boat conformation. Bond lengths and angles are normal.

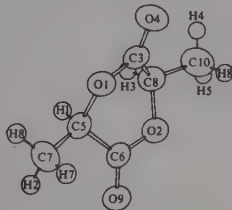


Fig. 1. The  $C_6H_8O_4$  molecule.

7,8-endo-EPOXY-Z-OXATRICYCLO[3.3.0.0<sup>4,6</sup>]OCTAN-3-ONE  
 $C_7H_6O_3$

P. MURRAY-RUST, R.C. GLEN, R.F. NEWTON and S.M. ALI, 1982. Acta Cryst., B38, 2704-2707.

Monoclinic,  $P2_1/c$ ,  $a = 6.36$ ,  $b = 9.58$ ,  $c = 9.71$  Å,  $\beta = 75.13^\circ$ ,  $D_m = 1.60$ ,  $Z = 4$ . Mo radiation,  $R = 0.041$  for 1124 reflexions.

In the molecule (Fig. 1) the cyclopentane ring is an envelope with the flap exo to the epoxide and the lactone ring is also an envelope. The  $O(10) \cdots C(3)$  distance is  $2.651(2)$  Å, which is unusually short for a non-bonded contact of this type and is in part due to the geometrical constraints imposed on the two five-membered rings by the  $C(4)-C(6)$  bond.  $C(3)$  is displaced from the plane of the carbonyl group by  $0.052(1)$  Å towards  $O(10)$ . The crystal structure consists of discrete molecules with no unusually short intermolecular contacts.

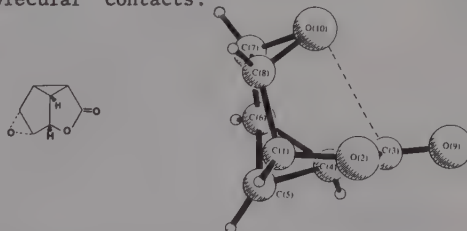


Fig. 1.  $C_7H_6O_3$ : perspective view of the molecule with the intramolecular  $O(10) \cdots C(3)$  interaction shown as a broken line.

3,6,10-TRIOXATETRACYCLO[7.1.0.0<sup>2,4</sup>.0<sup>5,7</sup>]DECANE  
 $C_7H_8O_3$  (I)

3-ACETYL-7,9-BIS(METHOXYCARBONYL)-3-AZATETRACYCLO[7.1.0.0<sup>2,4</sup>.0<sup>5,7</sup>]DECANE  
 $C_{15}H_{19}NO_5$  (II)

C. RUCKER, G. McMULLEN, C. KRUGER and H. PRINZBACH, 1982. Chem. Ber., 115, 2287-2308.

I. Monoclinic,  $P2_1/a$ ,  $a = 8.486$ ,  $b = 6.664$ ,  $c = 11.307$  Å,  $\beta = 77.39^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.059$  for 1097 reflexions.

II. Monoclinic,  $P2_1/n$ ,  $a = 8.657$ ,  $b = 9.108$ ,  $c = 19.201$  Å,  $\beta = 94.69^\circ$ ,  $Z = 4$ . Mo

radiation,  $R = 0.042$  for 1607 reflexions.

The structures were confirmed as those shown in Fig. 1. Major interplanar angles ( $^\circ$ ) are:

	I	II
C(7)C(8)C(9)/C(1)C(5)C(7)C(9)	38.7	36.2
C(1)C(5)C(7)C(9)/C(1)C(2)C(4)C(5)	56.3	58.5
C(1)O(3)C(9)/C(1)C(5)C(7)C(9)	105.2	107.8
(C(1)C(10)C(9))		
C(2)O(1)C(4)/C(1)C(2)C(4)C(5)	105.3	106.0
(C(2)N(1)C(4))		
C(5)O(2)C(7)/C(1)C(5)C(7)C(9)	104.8	110.7
(C(5)C(6)C(7))		

Transannular distances are:

C(1)...C(5)	3.065(4)	3.204(3)
C(7)...C(9)	2.452(4)	2.529(3)
O(2)...O(3)	4.603	

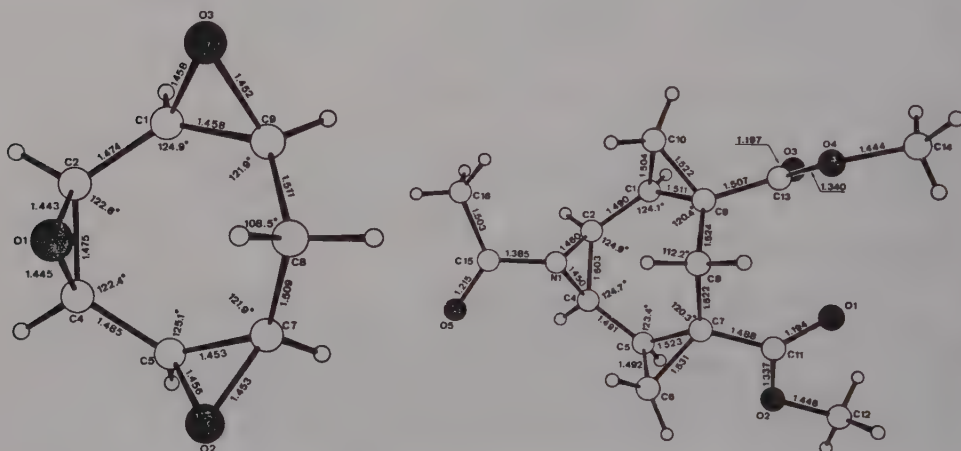


Fig. 1. The structures of  $C_7H_8O_3$  and  $C_{15}H_{19}NO_5$  with bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ).

#### TETRACHLOROPHTHALIC ANHYDRIDE

$C_8Cl_4O_3$  (I)

#### TETRABROMOPHTHALIC ANHYDRIDE

$C_8Br_4O_3$  (II)

D.S. SAKE GOWDA and R. RUDMAN, 1982. Acta Cryst., B38, 2842-2845 (for I and II).

T. UCHIDA, H. NAKANO and K. KOZAWA, 1982. Acta Cryst., B38, 2963-2965 (for I).

I. Monoclinic,  $P2_1/n$ ,  $a = 13.438$ ,  $b = 5.7874$ ,  $c = 12.332 \text{ \AA}$ ,  $\beta = 91.182^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.033$  for 1615 reflexions.

II. Monoclinic,  $P2_1/n$ ,  $a = 13.422$ ,  $b = 6.1794$ ,  $c = 12.680 \text{ \AA}$ ,  $\beta = 90.789^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.042$  for 1778 reflexions.

Both compounds have previously been studied using photographic data (1 and 2). In both molecules (Fig. 1) chemically equivalent but crystallographically unique bond lengths are equal, the benzene ring is slightly distorted, and the molecule is non-planar (the halogen atoms are out of the molecular plane and the plane of the benzene ring is inclined to the plane of the five-membered heterocyclic ring). The average C-Cl and C-Br distances are 1.719 and 1.878  $\text{\AA}$  (corrected values based on a rigid-body treatment) in I and II respectively.

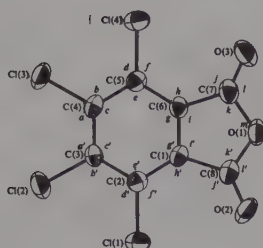


Fig. 1.  $C_8Cl_4O_3$  (I): perspective view of the molecule.

1. Structure Reports, 37B, 47.

2. Ibid, 41B, 88.

trans-5,6-DICYANO-7-OXABICYCLO[2.2.1]HEPT-2-ENE

$C_8H_6N_2O$

M. COSSU, R. VIANI, J. LAPASSET, J.P. AYCARD, C. MARFISI and H. BODOT, 1982. Cryst. Struct. Comm., 11, 1189-1192.

Monoclinic,  $P2_1/c$ ,  $a = 5.537$ ,  $b = 18.314$ ,  $c = 7.954$  Å,  $\beta = 120.00^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.047$  for 1161 reflexions.

In this molecule (Fig. 1), the  $C1=C2$  bond length is  $1.324(3)$  and  $C(4)-C(5)$   $1.561(3)$  Å. The two five-membered rings have slightly different puckering; the dihedral angles between the  $C3-O7-C6$  and  $C6-C1-C2-C3$  or  $C3-C4-C5-C6$  planes are  $130^\circ$  and  $118^\circ$  respectively.

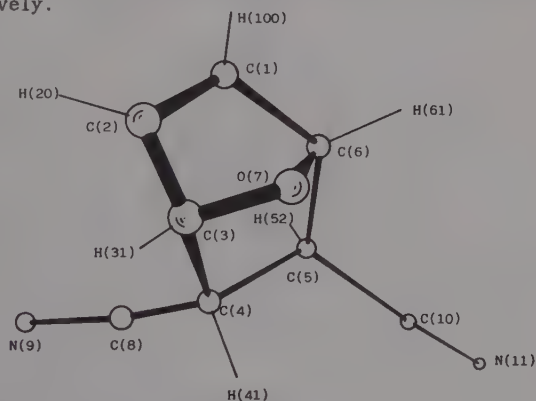


Fig. 1. The  $C_8H_6N_2O$  molecule.

5-HYDROXY-2(3H) BENZOFURANONE

$C_8H_6O_3$

G. BOCELLI and M.F. GRENIER-LOUSTALOT, 1982. J. Mol. Struct., 82, 301-306.

Monoclinic,  $P2_1/n$ ,  $a = 10.617$ ,  $b = 8.996$ ,  $c = 6.988$  Å,  $\beta = 76.5^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.047$  for 942 reflexions.

The molecules are held together in the crystal lattice by O-H...O hydrogen bonds (Fig. 1) with O...O  $2.78$  Å. The angle between the six- and five-membered ring planes is  $1.7^\circ$ .



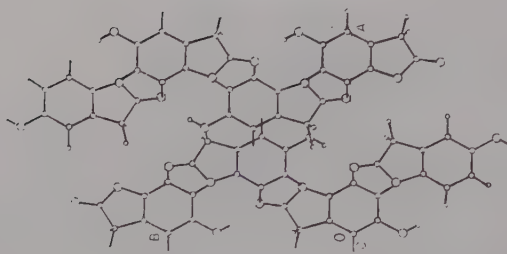


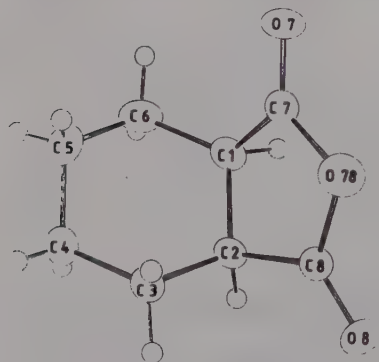
Fig. 1. The packing of  $C_8H_6O_3$ .

cis-1,2-CYCLOHEXANE-DICARBOXYLIC ACID ANHYDRIDE  
 $C_8H_{10}O_3$

A. PAWEL, K.-H. WOLLER, M. STRUMPEL and P. LUGER, 1982. Cryst. Struct. Comm., **11**, 647-652.

Orthorhombic,  $P2_12_12_1$ ,  $a = 14.382$ ,  $b = 7.874$ ,  $c = 6.769$  Å,  $Z = 4$ . Cu radiation,  $R = 0.054$  for 640 reflexions.

The cyclohexane ring (Fig. 1) adopts the expected chair conformation. Due to the anhydro bridge, the cyclohexane ring is flattened at C(1) and C(2). The five-membered anhydro ring takes up a slightly distorted envelope conformation with C(1) out of the plane through C(2), C(7), O(78) and C(8).



C(1) - C(2)	1.542(6)	C(4) - C(5)	1.523(7)
C(1) - C(6)	1.515(7)	C(5) - C(6)	1.505(8)
C(1) - C(7)	1.510(6)	C(7) - O(7)	1.212(5)
C(2) - C(3)	1.536(6)	C(7) - O(78)	1.395(6)
C(2) - C(8)	1.507(6)	C(8) - O(8)	1.219(6)
C(3) - C(4)	1.537(7)	C(8) - O(78)	1.379(5)

Fig. 1. The  $C_8H_{10}O_3$  structure and bond lengths.

3-exo-METHOXY-6,7-endo-EPOXY-2-OXABICYCLO[3.3.0]OCTANE  
 $C_8H_{12}O_3$

P. MURRAY-RUST, R.C. GLEN and R.F. NEWTON, 1982. Acta Cryst., **B38**, 2702-2704.

Monoclinic,  $C2/c$ ,  $a = 17.15$ ,  $b = 6.15$ ,  $c = 14.99$  Å,  $\beta = 82.47^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.0483$  for 905 reflexions.

The conformation of the bicyclic system is determined by the epoxy and methoxy substituents (Fig. 1). The cyclopentane ring adopts a shallow envelope conformation with the flap end to the epoxide (C(6)-C(5)-C(1)-C(8)  $10.5^\circ$ ). An approximate mirror plane passes through C(1) and O(9). The crystal structure consists of discrete molecules with no unusually short intermolecular contacts.

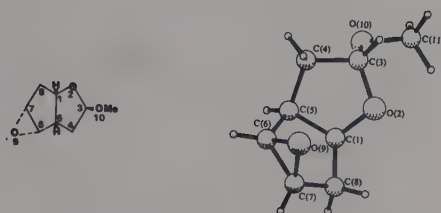


Fig. 1.  $C_8H_{12}O_3$ : a perspective view of the molecule.

#### TETRAIODOPHTHALIC ANHYDRIDE



D.S.S. GOWDA and R. RUDMAN, 1982. *J. Phys. Chem.*, **86**, 4356-4360.

Tetragonal,  $I4_1/a$ ,  $a = 22.655$ ,  $c = 9.136$  Å,  $D_m = 3.50$ ,  $Z = 16$ . Mo radiation,  $R = 0.037$  for 1512 reflexions.

In the molecule (Fig. 1), the average C-I distance is 2.087 Å and the iodine atoms are significantly displaced ( $-0.025$  to  $+0.109$  Å) from the least-squares plane through the benzene ring (with three iodine atoms on one side of the plane and one iodine atom on the other side). The angle between the six- and five-membered ring planes is  $1.47^\circ$ .

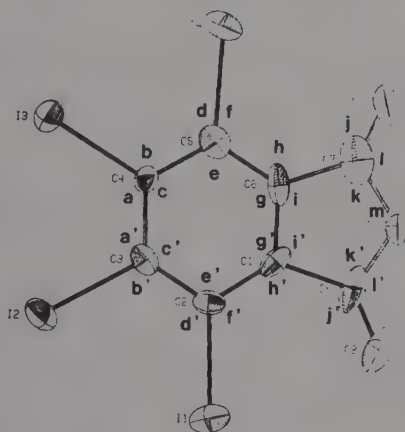


Fig. 1. A view of  $C_8I_4O_3$ .

#### 6-CHLORO-6-CYANO-1,5-DIMETHYL-3,8-DIOXATRICYCLO[3.2.1.0<sup>2,4</sup>]OCTANE $C_9H_{10}ClNO_2$

M. COSSU, R. VIANI, J. LAPASSET, J.P. AYCARD, C. MARFISI and H. BODOT, 1982. *Cryst. Struct. Comm.*, **11**, 1185-1188.

Orthorhombic,  $Pca2_1$ ,  $a = 10.784$ ,  $b = 8.438$ ,  $c = 10.865$  Å,  $Z = 4$ . Cu radiation,  $R = 0.061$  for 763 reflexions.

In the molecule (Fig. 1) the boat conformation of the six-membered ring is slightly distorted, with torsion angles  $C3-C2-C1-C6$   $-3.2(5)^\circ$  and  $C3-C4-C5-C6$   $4.1(5)^\circ$ . Bond lengths are as anticipated.

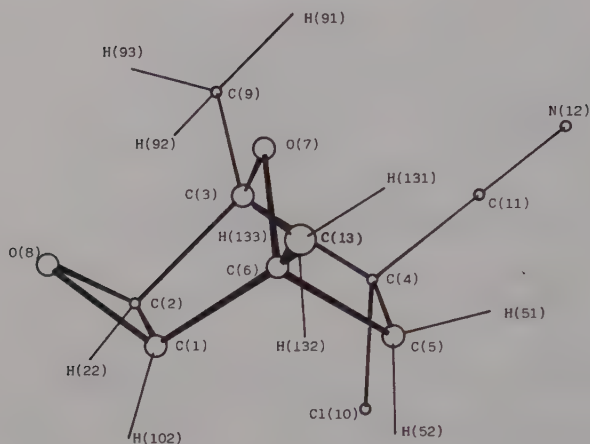


Fig. 1. A view of  $C_9H_{10}ClNO_2$ .

6-*exo*-HYDROXY-2-*exo*-METHYLBICYCLO[2.2.1]HEPTANE-2,6-CARBO-LACTONE  
 $C_9H_{12}O_3$

D.J. CHADWICK, W.B. SCHWEIZER, P. SEILER and S.N. WHITTLETON, 1982. *Acta Cryst.*, **B38**, 1043-1046.

Orthorhombic,  $Pbca$ ,  $a = 16.361$ ,  $b = 10.390$ ,  $c = 9.757$  Å,  $Z = 8$ . Mo radiation,  $R = 0.031$  for 1094 reflexions.

In the crystal structure (Fig. 1) the molecule exists as the closed hydroxy-lactone form. There are appreciable distortions from the idealized geometry which closely follow those found in the analogous decalin hydroxy-lactone (1).

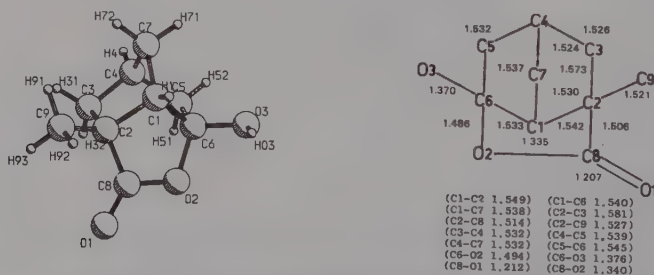


Fig. 1.  $C_9H_{12}O_3$ : a view of the molecule and bond lengths ( $\sigma$  0.0025 Å) corrected for libration.

1. Structure Reports, **45B**, 136.

1-OXOCYCLOHEXANE-2,6-DIONE-4-SPIRO-CYCLOPENTANE  
 $C_9H_{12}O_3$

G. BOCELLI, M.F. GRENIER-LOUSTALOT and Z. URBANCZYK-LIPKOWSKA, 1982. *J. Cryst. Spectrosc. Res.*, **12**, 407-414.

Orthorhombic,  $P2_12_12_1$ ,  $a = 17.540$ ,  $b = 7.457$ ,  $c = 6.661$  Å,  $Z = 4$ . Cu radiation,

$R = 0.072$  for 527 reflexions.

The two ring planes (Fig. 1) are almost perpendicular ( $88.9(3)^\circ$ ). The cyclopentane ring has a half-twist conformation and the six-membered ring is in half-chair conformation.

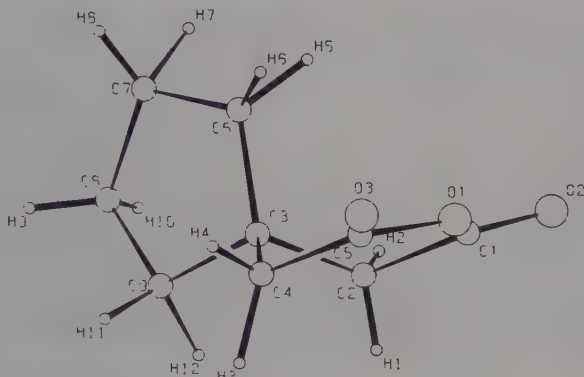


Fig. 1. A view of  $C_9H_{12}O_3$ .

#### 2-PHENOXY- $\gamma$ -BUTYROLACTONE

$C_{10}H_{10}O_3$

G. BOCELLI and M.-F. GRENIER-LOUSTALOT, 1982. *J. Mol. Struct.*, **82**, 295-299.

Monoclinic,  $P2_1/n$ ,  $a = 19.708$ ,  $b = 7.167$ ,  $c = 6.298$  Å,  $\beta = 84.2^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.053$  for 1397 reflexions.

The lactone ring has an envelope conformation (Fig. 1) and the two ring planes form a dihedral angle of  $133^\circ$ .

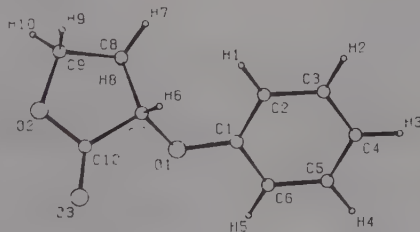


Fig. 1. The  $C_{10}H_{10}O_3$  molecule.

#### endo-3-CARBOXY-exo-5-IODOBICYCLO[2.2.2]OCTANE-2,6-CARBOLACTONE

$C_{10}H_{11}IO_4$  (I)

#### exo-3-METHOXYCARBONYL-endo-5-ACETOXYBICYCLO[2.2.2]OCTANE-2,6-CARBOLACTONE

$C_{13}H_{16}O_6$  (II)

R.M. CARMAN, S.S. SMITH, C.H.L. KENNARD, G. SMITH, A.H. WHITE and B.W. SKELTON, 1982. *Aust. J. Chem.*, **35**, 457-463.

I. Monoclinic,  $P2_1/c$ ,  $a = 7.192$ ,  $b = 23.785$ ,  $c = 13.199$  Å,  $\beta = 105.35^\circ$ ,  $D_m = 1.99$ ,  $Z = 8$ . Mo radiation,  $R = 0.033$  for 1918 reflexions.

II. Orthorhombic,  $P2_12_12_1$ ,  $a = 13.347$ ,  $b = 11.839$ ,  $c = 8.227$  Å,  $D_m = 1.38$ ,  $Z = 4$ . Mo radiation,  $R = 0.036$  for 630 reflexions.

The analysis confirmed the presence in both structures of  $\gamma$ -lactone bridges (Fig. 1). The bond lengths are normal and the conformations of both molecules are very similar. In I, molecules are linked into centrosymmetric dimers by O-H...O hydrogen bonds between the  $-\text{CO}_2\text{H}$  groups ( $2.61(1)$  Å).

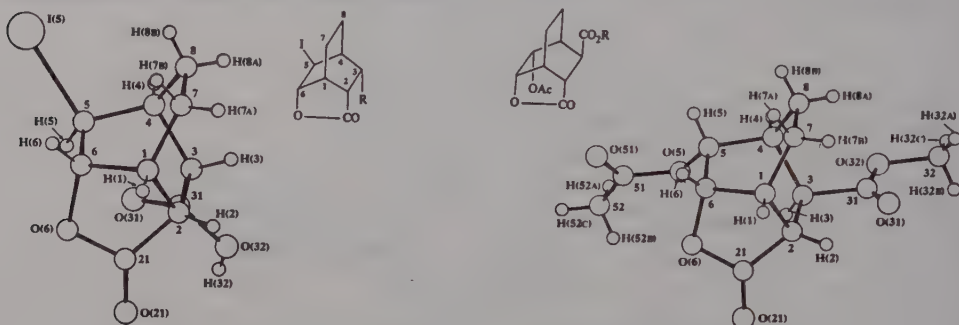


Fig. 1. Perspective views of  $\text{C}_{10}\text{H}_{11}\text{IO}_4$  (I) ( $\text{R} = \text{CO}_2\text{H}$ ) (left) and of  $\text{C}_{13}\text{H}_{16}\text{O}_6$  (II) ( $\text{R} = \text{Me}$ ) (right).

endo-2,endo-4-DIMETHYL-3-OXO-8-OXABICYCLO[3.2.1]OCT-6-ENE-exo-2-CARBONITRILE  
 $\text{C}_{10}\text{H}_{11}\text{NO}_2$

B. FOHLISCH, R. HERTER, E. WOLF, J.J. STEZOWSKI and E. ECKLE, 1982. Chem. Ber., **115**, 355-380.

Monoclinic,  $\text{P}2_1/\text{n}$ ,  $a = 6.236$ ,  $b = 14.395$ ,  $c = 10.151$  Å,  $\beta = 108.75^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.053$  for 2897 reflexions (at 120 K).

The structure was established as that shown in Fig. 1. Bond lengths and angles have normal values.

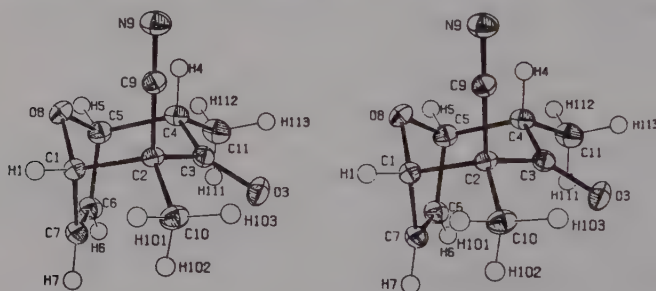
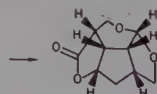


Fig. 1. Stereoview of  $\text{C}_{10}\text{H}_{11}\text{NO}_2$ .

(2aR,4S,4aS,5S,7aS,7bR)-OCTAHYDRO-2H,5H-1,4,7-TRIOXACYCLAPENT(j,k,l)-as-INDACEN-5-ONE  
 $\text{C}_{10}\text{H}_{12}\text{O}_4$



W.F. BERKOWITZ, S.C. CHOUDRY and J.F. HRABIE, 1982. J. Org. Chem., **47**, 824-829.

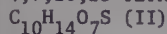
Trigonal,  $\text{P}3_1$  or  $\text{P}3_2$ ,  $a = 11.469$ ,  $c = 5.956$  Å,  $Z = 3$ . Cu radiation,  $R = 0.031$  for 578 reflexions.

The analysis confirmed the all-cis structure shown above.

## 1,4,7,10,13-PENTAOXACYCLOPENTADECANE-3,11,15-TRIONE



## 4,7,10,13-TETRAOXA-1-THIACYCLOPENTADECANE-3,11,15-TRIONE



Yu.G. GANIN, Yu.A. SIMONOV, N.G. LUK'YANENKO, Yu.A. POPKOV and A.V. BOGATSKII, 1982. Kristallografiya, 27, 916-922 [Sov. Phys. Crystallogr., 27, 550-553].

I. Orthorhombic,  $Pcab$ ,  $a = 16.865$ ,  $b = 18.322$ ,  $c = 7.758$  Å,  $Z = 8$ . Mo radiation,  $R = 0.050$  for 1655 reflexions.

II. Monoclinic,  $P2_1/b$ ,  $a = 15.508$ ,  $b = 9.252$ ,  $c = 9.016$  Å,  $\gamma = 76.26^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.079$  for 1933 reflexions.

The  $CH_2-CH_2$ , and  $CH_2-O$  bond lengths average 1.494 and 1.498, and 1.429 and 1.430 Å in I and II respectively (Fig. 1). Similarly, the  $O-C-C$  bond angles ( $110.7$  in I,  $109.1^\circ$  in II) and  $C-O-C$  bond angles ( $112.1$  in I and  $112.6^\circ$  in II) are almost the same in the two compounds. All the ester groups in I and II and the thioether group in II have planar structures owing to the partly double bond character of the  $C(=O)-O$  and  $C(=O)-S$  bonds.

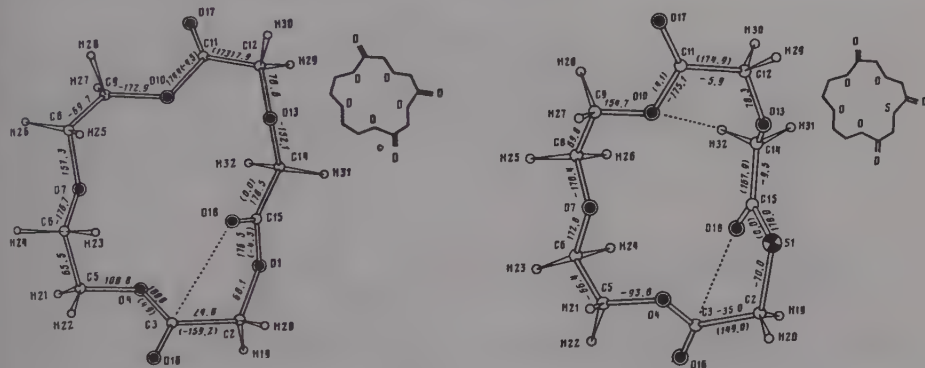


Fig. 1. The  $C_{10}H_{14}O_8$  (left) and  $C_{10}H_{14}O_7S$  (right) molecules.

## 7-ACETOXYCOUMARIN



V. RAMASUBBU, K. GNANAGURU, K. VENKATESAN and V. RAMAMURTHY, 1982. Canad. J. Chem., 60, 2159-2161.

Monoclinic,  $P2_1/c$ ,  $a = 3.833$ ,  $b = 22.665$ ,  $c = 10.975$  Å,  $\beta = 96.27^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.056$  for 1016 reflexions.

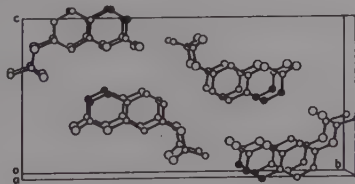


Fig. 1. The  $\beta$ -type packing of 7-acetoxycoumarin.



The dimerization of 7-acetoxycoumarin to yield a syn head-to-head dimer is rationalized by a knowledge of the crystal packing of the monomer (Fig. 1). There is so-called  $\beta$ -type packing, characterized by close packing of parallel and nearly planar reactive double bonds with a separation distance of 3.833 Å (the a-axis dimension).

### 3-PHENYL-1-OXACYCLOHEXANE-2,6-DIONE



G. BOCELLI, M.-F. GRENIER-LOUSTALOT, 1982. *Acta Cryst.*, **B38**, 2070-2072.

Monoclinic,  $P2_1/n$ ,  $a = 10.990$ ,  $b = 10.115$ ,  $c = 8.662$  Å,  $\beta = 107.1^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.085$  for 760 reflexions.

The non-aromatic ring has a half-boat conformation (Fig. 1) with C(4) 0.57(8) Å from the mean plane of the other ring atoms. Both carbonyl oxygens are equatorial and the C=O bonds are inclined at 96.2 and 100.7° to the normal to the ring plane. The angle between the mean planes of the two rings is 50.9(3)°.

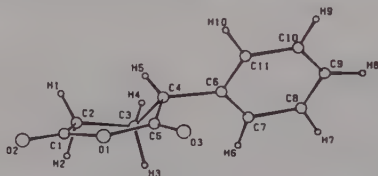
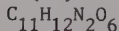


Fig. 1. The  $\text{C}_{11}\text{H}_{10}\text{O}_3$  molecule.

### 2-(2',5'-DINITROPHENOXY)TETRAHYDROPYRAN



P.G. JONES, G.M. SHELDRICK, A.J. KIRBY and R. GLENN, 1982. *Z. Krist.*, **161**, 253-258.

Monoclinic,  $P2_1/n$ ,  $a = 7.940$ ,  $b = 7.911$ ,  $c = 19.455$  Å,  $\beta = 98.34^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.050$  for 1542 reflexions.

The molecule is shown in Fig. 1. The exocyclic acetal bond length (C(1)-O(1) 1.458(3) Å) is within half an e.s.d. of that predicted by a bond length/reactivity correlation study. The C(1)-O(10) distance is 1.383(4) Å.

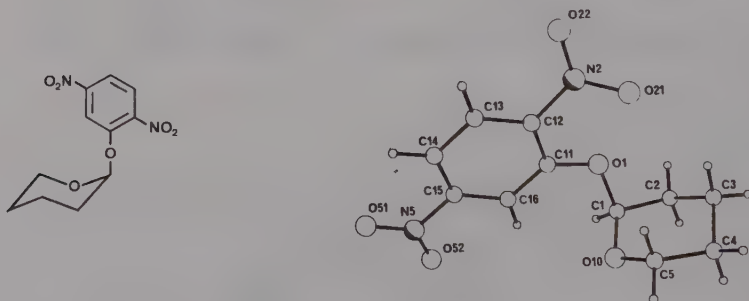
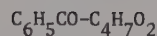
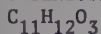


Fig. 1. A view of  $\text{C}_{11}\text{H}_{12}\text{N}_2\text{O}_6$ .

## 5-BENZOYL-1,3-DIOXANE



R. ROQUES, M. DELMAS and A. GASET, 1982. Cryst. Struct. Comm., 11, 751-754.

Orthorhombic,  $P2_1ab$ ,  $a = 19.458$ ,  $b = 16.283$ ,  $c = 6.229$  Å,  $Z = 8$ . Mo radiation,  $R = 0.061$  for 583 reflexions.

The analysis shows that the benzoyl group is in an equatorial position on the 1,3-dioxane ring which is in chair conformation.

## 7,8-DIHYDRO-4,7,7-TRIMETHYL-2H,5H-PYRANO[4,3-b]PYRAN-2,5-DIONE



N.G. CHARLES, E.A.H. GRIFFITH, C.W. MURTIASHAW, S.D. BURKE and E.L. AMMA, 1982. Acta Cryst., B38, 2496-2498.

Monoclinic,  $P2_1/c$ ,  $a = 11.908$ ,  $b = 9.611$ ,  $c = 9.437$  Å,  $\beta = 107.99^\circ$ ,  $D_m = 1.34$ ,  $Z = 4$ . Mo radiation,  $R = 0.043$  for 1095 reflexions.

In the molecule (Fig. 1) the ring system containing O(1) is planar including the exocyclic atoms but the remainder of the molecule is considerably out of this plane. There is considerable charge delocalization within the O(1) ring and this results in the relatively short C(9)-O(1) distance of 1.356(4) Å. The C(5)-O(6) distance of 1.347(4) Å is short because of geometrical factors but other bond lengths and angles are normal. The molecules are separated in the crystal by van der Waals distances.

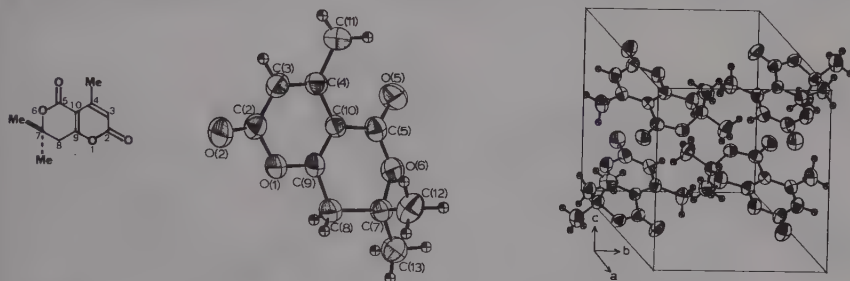


Fig. 1.  $\text{C}_{11}\text{H}_{12}\text{O}_4$ : views of the molecule and of the unit cell.

## 2-(4'-CHLOROPHENOXY)TETRAHYDROPYRAN



P.G. JONES, G.M. SHELDRIK, A.J. KIRBY, R. GLENN, P. RAMASWAMY and M. HALSTENBERG, 1982. Z. Krist., 159, 265-270.

Triclinic,  $P\bar{1}$ ,  $a = 6.774$ ,  $b = 8.900$ ,  $c = 9.811$  Å,  $\alpha = 66.06$ ,  $\beta = 86.67$ ,  $\gamma = 76.87^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.040$  for 1562 reflexions.

The C-O bond lengths at the acetal centre C(1) are C(1)-O(1) 1.427(4) and C(1)-O(10) 1.398(3) Å (Fig. 1) and are in accord with expected values.

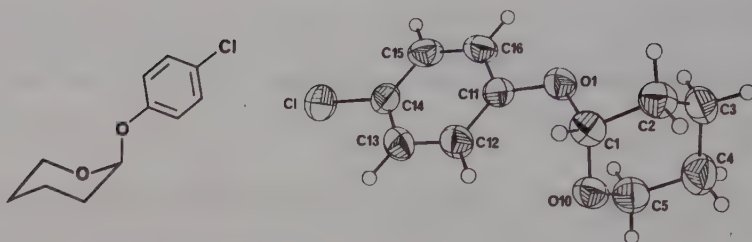


Fig. 1. The  $C_{11}H_{13}ClO_2$  molecule.

ACETILCYCLOPENTADIENYL-SODIUM TETRAHYDROFURANATE  
 $C_{11}H_{15}NaO_2$



R.D. ROGERS, J.L. ATWOOD, M.D. RAUSCH, D.W. MACOMBER and W.P. HART, 1982. *J. Organometal. Chem.*, **238**, 79-85.

Monoclinic,  $P2_1/c$ ,  $a = 6.698$ ,  $b = 16.095$ ,  $c = 10.661$  Å,  $\beta = 92.93^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.080$  for 661 reflexions.

The sodium atom is coordinated through O(1) to two  $C_5H_4CMeO$  units forming a dimer (Fig. 1). One THF ligand is coordinated to each Na atom, and the dimers are linked in a polymeric chain through the ionic contact between the Na atoms and the cyclopentadienyl groups (Fig. 1). The two Na-O(1) distances are 2.329 and 2.295 Å and the Na-O(2) distance is 2.354 Å. The Na-C(cyclopentadienyl) distances range from 2.69 to 2.94 Å and the O(1)-Na-O(1) angle is  $84.7^\circ$ .

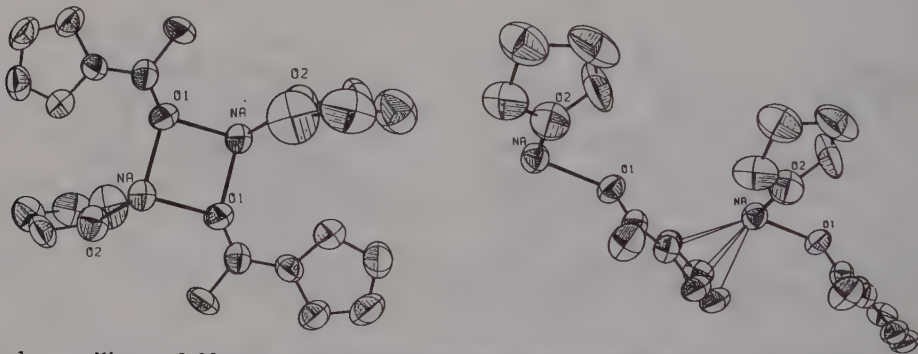


Fig. 1. Views of  $\{[C_5H_4CMeO]Na.THF\}_n$  showing the dimeric unit (left) and the polymerization of the dimeric units (right).

(1R,2S,3S)-1,2-EPOXY-3-METHYL-4,4-DIOXYETHYLENECYCLOHEXANE-1-CARBOXYLIC ACID METHYL ESTER  
 $C_{11}H_{16}O_5$

J.H. NOORDIK and J.M.M. SMITS, 1982. *Cryst. Struct. Comm.*, **11**, 1107-1110.

Monoclinic,  $P2_1/c$ ,  $a = 8.596$ ,  $b = 10.116$ ,  $c = 12.959$  Å,  $\beta = 89.13^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.068$  for 1588 reflexions.

The analysis establishes the relative configuration at the chiral centres as shown in Fig. 1. Geometrical parameters have normal values.

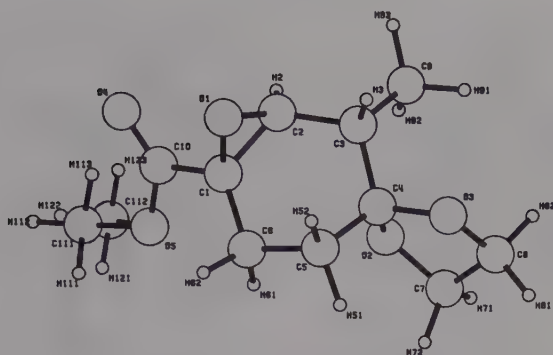


Fig. 1. A view of  $C_{11}H_{16}O_5$ .

trans-2-t-BUTYL-7-OXABICYCLO[4.1.0]HEPTANE-3-trans-CARBONITRILE  
 $C_{11}H_{17}NO$

N. EBBY, J. LAPASSET, L. PIZZALA and H. BODOT, 1982. Acta Cryst., B38, 3128-3130.

Monoclinic,  $P2_1/c$ ,  $a = 11.47$ ,  $b = 6.67$ ,  $c = 16.10$  Å,  $\beta = 120.4^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.071$  for 1354 reflexions.

The six-membered ring (Fig. 1) is in a half-chair conformation with some puckering.

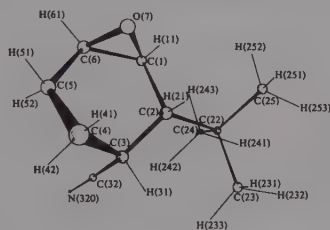


Fig. 1. The  $C_{11}H_{17}NO$  molecule.

2,3,4,4,5,6-HEXACHLORO-12-OXOPENTACYCLO[5.4.1.1<sup>8,11</sup>.0<sup>3,10</sup>.0<sup>5,9</sup>]TRIDECANE  
 $C_{12}H_8Cl_6O$

J.W. APSIMON, K. YAMASAKI, A. FRUCHIER, A.S. CHAU and C.P. HUBER, 1982. Canad. J. Chem., 60, 501-508.

Monoclinic,  $P2_1/c$ ,  $a = 13.680$ ,  $b = 8.515$ ,  $c = 25.963$  Å,  $\beta = 115.71^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.039$  for 3383 reflexions.

There are two independent molecules in the asymmetric unit (Fig. 1) with essentially identical geometries. The analysis confirms a structural assignment made from spectroscopic data. The five-membered rings are all in envelope conformation and the six-membered ring is in the boat form.

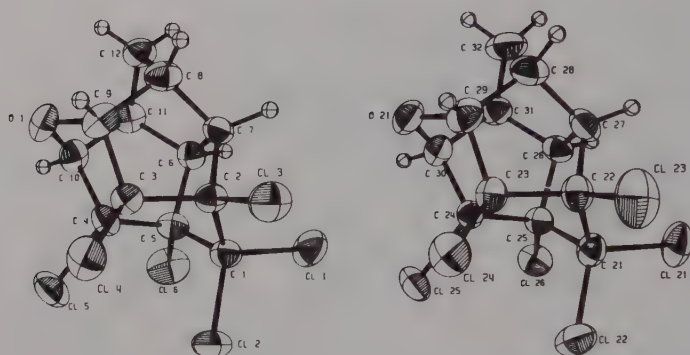


Fig. 1. Views of the two independent molecules of  $C_{12}H_8Cl_6O$ .

2-BROMO-6,7-EPOXY-cis-3a,7a-DIHYDROINDEN-1-ONE ETHYLENE KETAL 4-CARBOXALDEHYDE  
 $C_{12}H_{11}BrO_4$

J.H. NOORDIK, P.T.G. SILLEKENS and A.J.H. KLUNDER, 1982, *Cryst. Struct. Comm.*, **11**, 1709-1712.

Monoclinic,  $P2_1/n$ ,  $a = 8.039$ ,  $b = 9.352$ ,  $c = 15.145$  Å,  $\beta = 91.16^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.037$  for 934 reflexions.

The analysis establishes the stereochemistry of this reaction product to be as shown in Fig. 1.

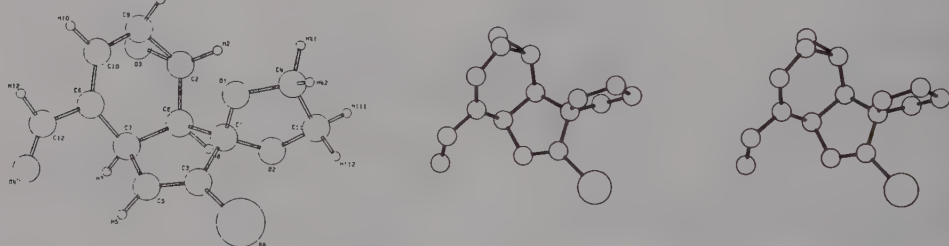


Fig. 1. A view of the  $C_{12}H_{11}BrO_4$  molecule showing the labelling and a stereoview.

1,4,7,10,13,16-HEXAOXACYCLOOCTADECANE LITHIUM PERCHLORATE DIHYDRATE

$C_{12}H_{24}ClLiO_{10}, 2H_2O$  (I)

$LiClO_4, (CH_2CH_2O)_6, 2H_2O$

1,4,7,10,13,16-HEXAOXACYCLOOCTADECANE BIS(LITHIUM THIOCYANATE) DIHYDRATE

$C_{14}H_{24}Li_2N_2O_6S_2, 2H_2O$  (II)

$(LiSCN)_2(CH_2CH_2O)_6, 2H_2O$

P. GROTH, 1982. *Acta Chem. Scand.*, **A36**, 109-115.

I. Monoclinic,  $P2_1/c$ ,  $a = 17.006$ ,  $b = 7.058$ ,  $c = 20.849$  Å,  $\beta = 128.72^\circ$ ,  $D_m = 1.35$ ,  $Z = 4$ . Mo radiation,  $R = 0.054$  for 2451 reflexions (at  $-150^\circ C$ ).

II. Monoclinic,  $P2_1/n$ ,  $a = 9.533$ ,  $b = 16.252$ ,  $c = 13.855$  Å,  $\beta = 100.49^\circ$ ,  $D_m = 1.30$ ,  $Z = 4$ . Mo radiation,  $R = 0.030$  for 3019 reflexions (at  $-150^\circ C$ ).

The cyclic hexaether of the perchlorate complex (I) adopts the non-regular  $D_{3d}$  conformation whilst that of the (2:1) thiocyanate complex is the bi-angular [8,10] conformation (Fig. 1). Three O atoms of the  $ClO_4^-$  ion are disordered and accept hydrogen bonds from the two water molecules. In addition, two of the crown ether O

atoms serve as hydrogen bond acceptors. The  $\text{Li}^+$  cation is coordinated to both water molecules and two ring O atoms: Li-OW(1) 1.922, Li-OW(2) 1.906, Li-O(3) 2.070, Li-O(4) 2.124 Å. In (II) each Li has fourfold coordination. One is coordinated to two ether O atoms and two water molecules: Li(1)-OW(1) 1.966, Li(1)-OW(2) 1.899, Li(1)-O(3) 2.073, and Li(1)-O(4) 1.995 Å. The other is coordinated to one ether O atom, one water molecule and two N atoms from the thiocyanate ions: Li(2)-O(2) 1.991, Li(2)-OW(1) 1.996, Li(2)-N(1) 2.011 and Li(2)-N(2) 1.998 Å.

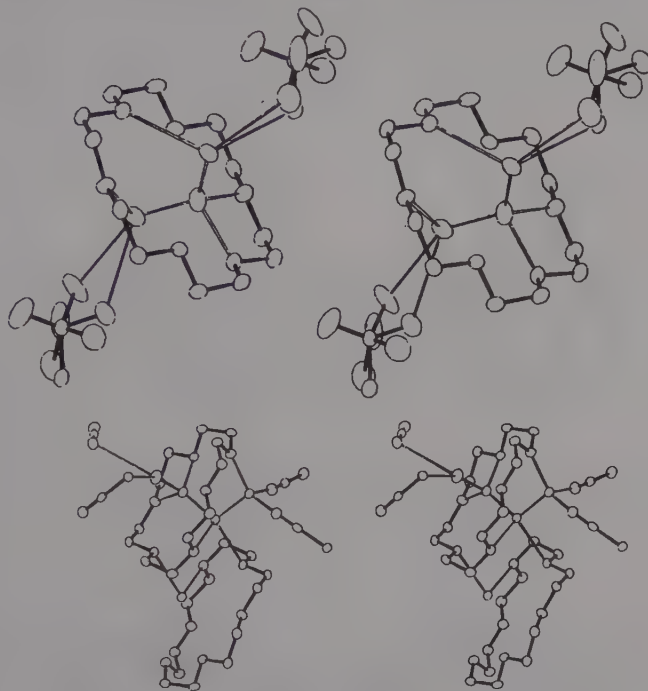


Fig. 1. Stereoscopic views of  $\text{C}_{12}\text{H}_{24}\text{O}_6, \text{LiClO}_4, 2\text{H}_2\text{O}$  (top), and  $\text{C}_{12}\text{H}_{24}\text{O}_6, (\text{LiSCN})_2, 2\text{H}_2\text{O}$  (bottom).

1,4,7,10,13,16-HEXAOXACYCLOOCTADECANE HYDRAZINIUM PERCHLORATE

$\text{C}_{12}\text{H}_{29}\text{ClN}_2\text{O}_{10}$  (I)

$\text{C}_{12}\text{H}_{24}\text{O}_6, \text{N}_2\text{H}_5^+ \text{ClO}_4^-$

1,4,7,10,13,16-HEXAOXACYCLOOCTADECANE HYDROXYLAMMONIUM PERCHLORATE WATER

$\text{C}_{12}\text{H}_{28}\text{ClNO}_{10}, 0.5(\text{H}_2\text{O})$  (II)

$\text{C}_{12}\text{H}_{24}\text{O}_6, \text{NH}_4\text{O}^+ \text{ClO}_4^-, 0.5(\text{H}_2\text{O})$

1,4,7,10,13,16-HEXAOXACYCLOOCTADECANE METHYLAMMONIUM PERCHLORATE

$\text{C}_{13}\text{H}_{30}\text{ClNO}_{10}$  (III)

$\text{C}_{12}\text{H}_{24}\text{O}_6, \text{CH}_3\text{NH}_3^+ \text{ClO}_4^-$

K.N. TRUEBLOOD, C.B. KNOBLER, D.S. LAWRENCE and R.V. STEVENS, 1982. J. Am. Chem. Soc., 104, 1355-1362.

I. Orthorhombic,  $\text{Pn}2_1\text{a}$ ,  $a = 14.657$ ,  $b = 13.652$ ,  $c = 9.244$  Å,  $Z = 4$ . Mo radiation,  $R = 0.057$  for 3629 reflexions (at 115 K).

II. Monoclinic,  $\text{C}2/c$ ,  $a = 19.410$ ,  $b = 10.231$ ,  $c = 21.321$  Å,  $\beta = 116.71^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.066$  for 5512 reflexions (at 115 K).

III. Monoclinic,  $\text{C}2/c$ ,  $a = 24.518$ ,  $b = 8.594$ ,  $c = 21.447$  Å,  $\beta = 117.04^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.086$  for 2991 reflexions.



The macrocycle in each of these structures is in the ideal crown conformation, with all C-C bonds gauche and all C-O bonds trans. Complex I is unusual in that the  $\text{-NH}_3^+$  nitrogen atom lies close to the median plane of the 6 oxygen of the crown ring, in a nesting position 0.11 Å from this plane. It is H-bonded to the lower triangle of O atoms; the  $\text{-NH}_2$  group is H-bonded to the upper triangle in the host, one of these being weakly bifurcated, involving a  $\text{ClO}_4^-$  oxygen as well. In II the nitrogen of the  $\text{-NH}_3^+$  group lies 0.68 Å from the plane of the O atoms of the macrocycle; the H atoms are involved in bifurcated H-bonds, each to two adjacent O atoms. The OH group of the  $\text{NH}_3\text{OH}^+$  cation is H-bonded to a water molecule that lies on a twofold axis; the water molecule is in turn H-bonded to two  $\text{ClO}_4^-$  anions. In III, the  $\text{-NH}_3^+$  nitrogen lies 0.83 Å above the plane of the ring O atoms, perching on the upper triangle of O atoms.

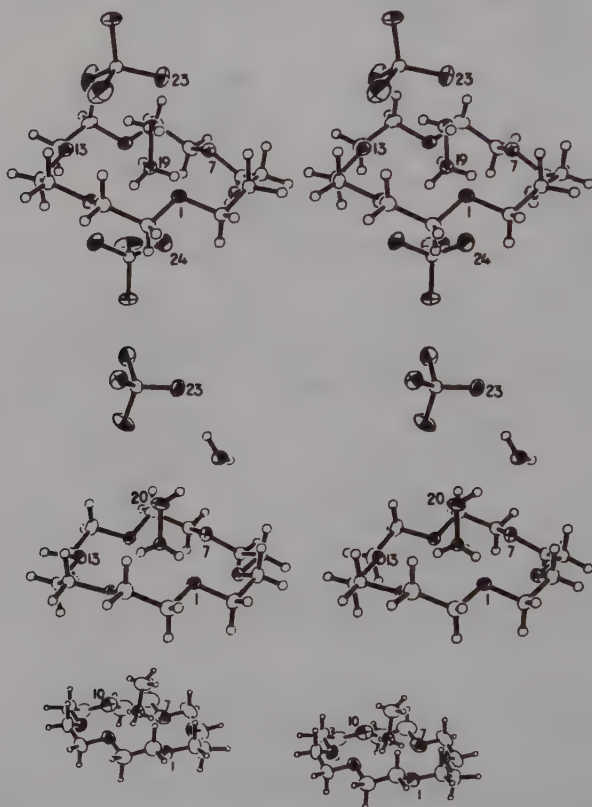
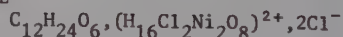
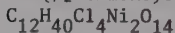


Fig. 1. Stereoviews of  $\text{C}_{12}\text{H}_{24}\text{O}_6, \text{N}_2\text{H}_5^+\text{ClO}_4^-$  (top),  $\text{C}_{12}\text{H}_{24}\text{O}_6, \text{NH}_3\text{OH}^+\text{ClO}_4^-, 0.5\text{H}_2\text{O}$  (middle), and  $\text{C}_{12}\text{H}_{24}\text{O}_6, \text{CH}_3\text{NH}_3^+$  (bottom).

BIS( $\mu_2$ -CHLORO)OCTAAQUADINICKEL(II) 18-CROWN-6 DICHLORIDE



J. JARRIN, F. DAWANS, F. ROBERT and Y. JEANNIN, 1982. *Polyhedron*, **1**, 409-412.

Triclinic,  $\text{P}\bar{1}$ ,  $a = 8.828$ ,  $b = 9.693$ ,  $c = 10.616$  Å,  $\alpha = 55.74$ ,  $\beta = 63.47$ ,  $\gamma = 63.62^\circ$ ,  $Z = 1$ . Mo radiation,  $R = 0.045$  for 1301 reflexions.

Both crown ether and  $[\text{Ni}_2\text{Cl}_2(\text{H}_2\text{O})_8]^{2+}$  moieties lie on inversion centres (Fig. 1). The Ni-coordination is octahedral, with Ni-Cl 2.403 and 2.420, Ni-O 2.050-2.076(7) Å.

The crown ether oxygen atoms hydrogen bond with the coordinated water molecules and the crown conformation is not regular.

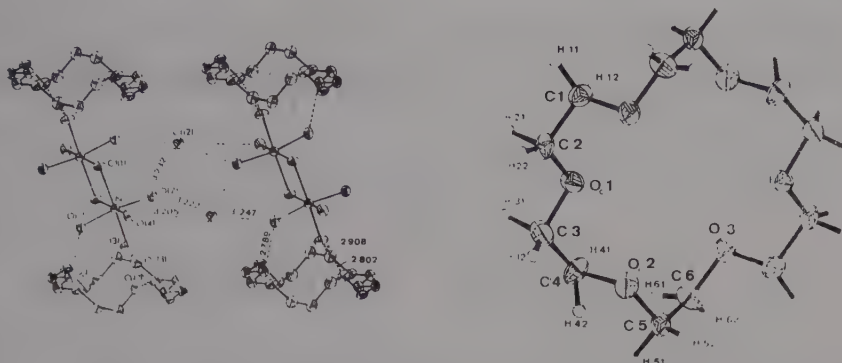
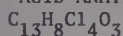


Fig. 1. The hydrogen bonding in  $C_{12}H_{40}Cl_4Ni_2O_{14}$  and the 18-crown-6 conformation.

exo-endo-3,4,5,6-TETRACHLOROTRICYCLO[6.2.1.0<sup>2,7</sup>]UNDECA-3,5-DIENE 9,10-DICARBOXYLIC  
ACID ANHYDRIDE



A.N. SHNULIN, S.A. ISMAILOV, M.S. SALAKHOV, Kh.S. MAMEDOV, T.Kh. KURBANOV and M.M. GUSEINOV, 1982. Kristallografiya, 27, 273-278 [Sov. Phys. Crystallogr., 27, 167-170].

Monoclinic,  $P2_1/a$ ,  $a = 10.575$ ,  $b = 9.559$ ,  $c = 14.028$  Å,  $\beta = 94.007^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.061$  for 1100 reflexions.

The bond lengths and angles in the molecule (Fig. 1) are similar to those in the exo-exo isomer (1). The hexadiene ring is planar within experimental error and the dihedral angle between it and the plane fragment C(1),C(2),C(7),C(8) is  $118.6^\circ$ . The dihedral angle between this fragment and the best plane through C(1),C(8),C(9),C(10) is  $114.4^\circ$ , and the angle between the anhydride ring (C(9),C(10),C(12),C(13),O(1)) and the C(1),C(8),C(9),C(10) fragment is  $123.7^\circ$ .

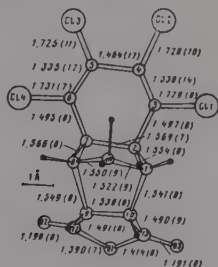


Fig. 1. Bond lengths in  $C_{13}H_8Cl_4O_3$ .

1. Structure Reports, 48B, 356.

XANTHONE



S.C. BISWAS and R.K. SEN, 1982. Ind. J. Pure Appl. Phys., 20, 414-415.

Orthorhombic,  $P2_12_12_1$ ,  $a = 13.63$ ,  $b = 14.11$ ,  $c = 4.86$  Å,  $D_m = 1.382$ ,  $Z = 4$ . Cu radiation,  $R = 0.133$  for 680 reflexions.

The two halves of the xanthone molecule (Fig. 1) along the  $O(15)=C(10)-O(14)$  axis make an angle of  $3^\circ$  with each other, and the dihedral angle between the planes of the benzene rings is  $3^\circ$ . Distances within the benzene rings range from 1.33 to 1.43 Å. Other distances are:  $C(3)-O(14)$  1.36,  $C(4)-O(14)$  1.33,  $C(9)-C(10)$  1.47,  $C(11)-C(10)$  1.42, and  $C(10)-O(15)$  1.25 Å.

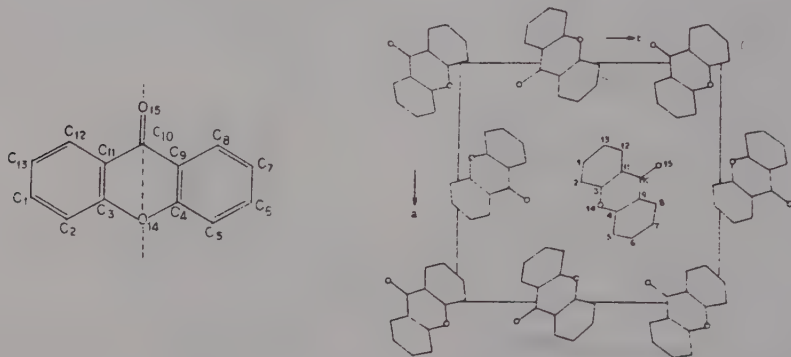


Fig. 1. Schematic structure of  $C_{13}H_8O_2$  (left), and the molecular packing viewed along the  $c$  axis.

#### 2-NITRO-7-METHOXYNAPHTHO[2,1-b]FURAN

$C_{13}H_9NO_4$

G. BRAVIC, J.P. BIDEAU and C. COURSEILLE, 1982. Cryst. Struct. Comm., **11**, 409-412.

Monoclinic,  $P2_1/c$ ,  $a = 3.851$ ,  $b = 26.957$ ,  $c = 10.547$  Å,  $\beta = 98.38^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.032$  for 750 reflexions.

The molecule (Fig. 1) is almost planar. Crystal cohesion involves C-H...O hydrogen bonds ( $H...O$  2.44(3) and 2.55(3) Å) and donor-acceptor  $\pi$ -interactions between molecules translated along the  $a$ -direction (interplanar spacing 3.44 Å).

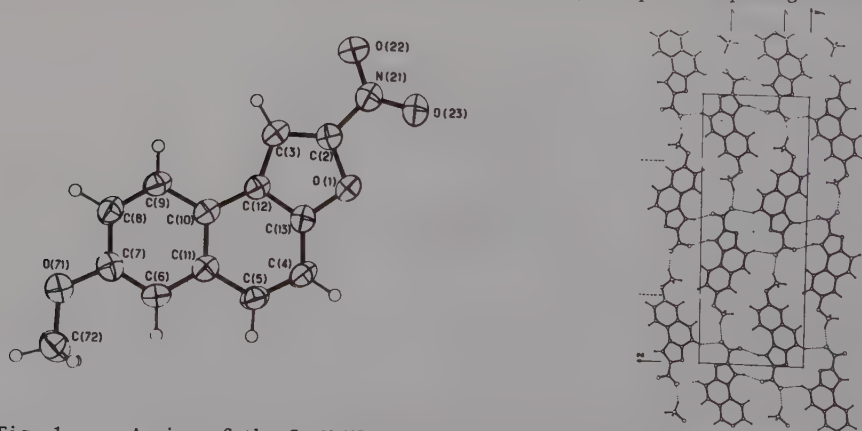
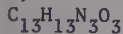


Fig. 1. A view of the  $C_{13}H_9NO_4$  molecule and a packing diagram viewed along the  $a$ -direction.

## 2,3-endo-EPOXYBICYCLO[3.2.0]HEPTAN-6-ONE p-NITROPHENYL-HYDRAZONE



P. MURRAY-RUST, R.C. GLEN and R.F. NEWTON, 1982. *Acta Cryst.*, B38, 2698-2700.

Monoclinic,  $P2_1/c$ ,  $a = 9.50$ ,  $b = 13.73$ ,  $c = 9.70$  Å,  $\beta = 83.19^\circ$ ,  $D_m = 1.37$ ,  $Z = 4$ . Mo radiation,  $R = 0.0479$  for 1538 reflexions.

In the molecule (Fig. 1) the cyclopentane epoxide fragment is in the endo envelope conformation with the possibility of a transannular  $\text{O} \cdots \text{C}=\text{N}$  interaction, with  $\text{O}(8) \cdots \text{C}(6)$  2.990(3) Å. The molecules are linked into chains in the crystal by hydrogen bonds, with  $\text{N}(10) \cdots \text{O}(8')$  2.970(2) Å.

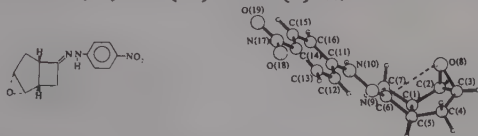
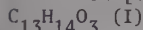
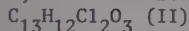
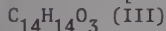
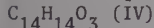
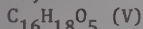


Fig. 1.  $\text{C}_{13}\text{H}_{13}\text{N}_3\text{O}_3$ : the conformation of the molecule with the intramolecular  $\text{O}(8) \cdots \text{C}(6)$  interaction shown as a broken line.

TETRACYCLO[4.4.0.1<sup>1,4</sup>.1<sup>7,10</sup>]DODEC-5-ENE-2,3-DIYL CARBONATE2,3-DICHLOROTETRACYCLO[4.4.0.1<sup>1,4</sup>.1<sup>7,10</sup>]DODEC-5-ENE-2,3-DIYL CARBONATETETRACYCLO[4.4.0.1<sup>1,4</sup>.1<sup>7,10</sup>]DODEC-5-ENE-2,3-exo-DICARBOCYCLIC ANHYDRIDETETRACYCLO[4.4.0.1<sup>1,4</sup>.1<sup>7,10</sup>]DODEC-5-ENE-2,3-endo-DICARBOXYLIC ANHYDRIDE

## 4a,8a-EPOXY-2,3-BIS(METHOXYCARBONYL)SESQUINORBORN-2-ENE



R. SUBRAMANYAM, P.D. BARTLETT, G.Y.M. IGLESIAS, W.H. WATSON and J. GALLOY, 1982. *J. Org. Chem.*, 47, 4491-4498.

I. Orthorhombic,  $Pbca$ ,  $a = 11.609$ ,  $b = 15.621$ ,  $c = 11.406$  Å,  $Z = 8$ . Cu radiation,  $R = 0.038$  for 837 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 7.638$ ,  $b = 19.714$ ,  $c = 8.872$  Å,  $\beta = 111.91^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.061$  for 1214 reflexions.

III. Monoclinic,  $C2/c$ ,  $a = 24.641$ ,  $b = 6.5765$ ,  $c = 12.076$  Å,  $\beta = 98.374^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.063$  for 1539 reflexions.

IV. Monoclinic,  $P2_1/c$ ,  $a = 14.016$ ,  $b = 6.5765$ ,  $c = 12.076$  Å,  $\beta = 98.374^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.062$  for 976 reflexions.

V. Orthorhombic,  $Pbca$ ,  $a = 26.059$ ,  $b = 11.132$ ,  $c = 9.542$  Å,  $Z = 8$ . Cu radiation,  $R = 0.029$  for 1160 reflexions.

Compounds I and II (Fig. 1) are similar in structure, with slight differences in interplanar angles imposed by the replacement of two hydrogen atoms by chlorine. The norbornane systems in I, II, III and IV are essentially identical. In V, intermolecular packing interactions involving the ester side chains lead to a twist of  $\text{C11-C2-C3-C13}$  from coplanarity (torsion angle  $4.9^\circ$ ).

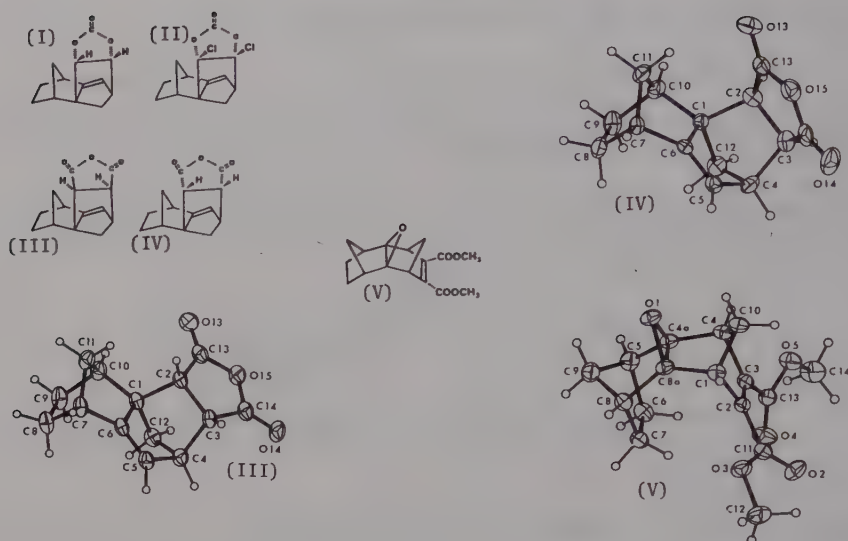


Fig. 1. The norbornane containing systems I - V.

METHYL 6-OXO-2 $\alpha$ ,3,4,5,5 $\alpha$ ,6,7,8,8 $\alpha$ ,8 $\beta\alpha$ -DECAHYDRO-2H-NAPHTHO[1,8-bc]FURAN-8 $\beta\alpha$ -CARBOXYLATE  
 $C_{13}H_{18}O_4$

B. RUBIN, J. EPSTEIN and L. BROOKS, 1982. *Acta Cryst.*, **B38**, 2959-2961.

Monoclinic,  $P2_1/c$ ,  $a = 8.039$ ,  $b = 14.275$ ,  $c = 11.730$  Å,  $\beta = 118.04^\circ$ ,  $D_m = 1.35$ ,  $Z = 4$ . Mo radiation,  $R = 0.053$  for 1765 reflexions.

In the molecule (Fig. 1) the average C-C bond length for the decalin system is 1.524 Å, in close agreement with the expected value. The six-membered ring containing C(7) is in the highly strained twisted-boat conformation, while the C(1)-C(6) ring is in the more stable chair conformation.

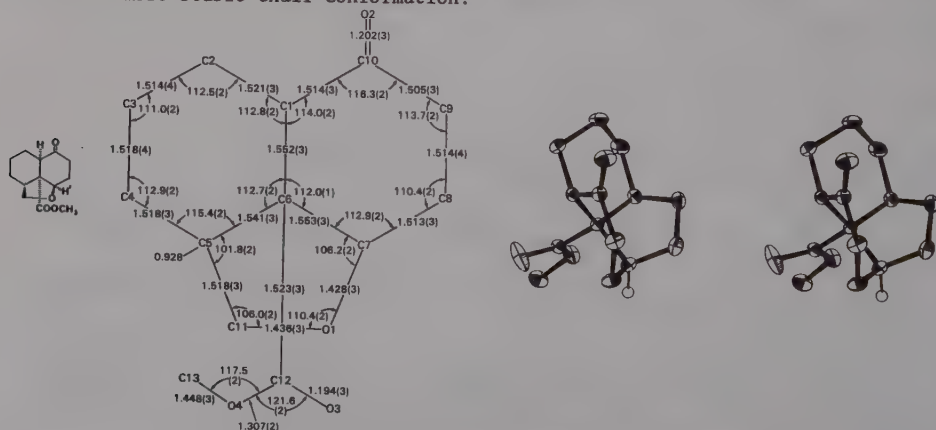
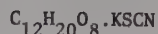


Fig. 1.  $C_{13}H_{18}O_4$ : bond lengths and angles and a stereoscopic view of the molecule.

1,4,7,10,13,16-HEXAOXACYCLOOCTADECANE-2,6-DIONE POTASSIUM THIOCYANATE  
 $C_{13}H_{20}KNO_8S$



S.B. LARSON and N.K. DALLEY, 1982. *Acta Cryst.*, **B38**, 1309-1312.

Monoclinic,  $P2_1/c$ ,  $a = 12.517$ ,  $b = 10.943$ ,  $c = 15.145$  Å,  $\beta = 119.975^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.046$  for 2675 reflexions.

In the complex (Fig. 1) the cation is situated in the cavity of the polyether ring, coordinated to the six O atoms of the ring (the average K-O distance is 2.75(5) Å) and to two anions (with K-N 2.873 and 2.939, K-S 3.355 and 3.124 Å). Atoms O(7), C(8), C(9) and O(10) of the polyether ring and the atoms of the anion are disordered.

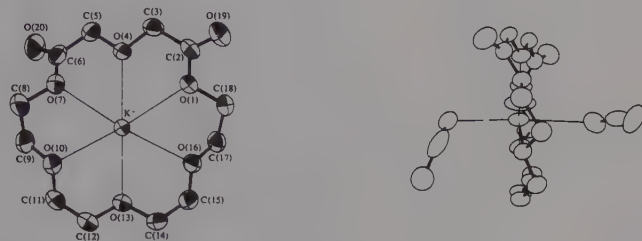


Fig. 1.  $C_{12}H_{20}O_8 \cdot KSCN$ : views of the complex; the  $SCN^-$  anion is omitted from the drawing on the left.

13-METHYL-2,10-DIOXATRICYCLO[4.4.4.0<sup>1,6</sup>]TETRADECANE  
 $C_{13}H_{22}O_2$

K.L. BROWN, G.J. DOWN, J.D. DUNITZ and P. SEILER, 1982. *Acta Cryst.*, **B38**, 1241-1245.

Triclinic,  $P\bar{1}$ ,  $a = 6.347$ ,  $b = 8.685$ ,  $c = 11.336$  Å,  $\alpha = 68.47$ ,  $\beta = 82.37$ ,  $\gamma = 80.09^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.033$  for 4080 reflexions (at 96 K).

The molecule (Fig. 1) adopts the chair-chair form and this results in the chemically equivalent C(6)-O(5) and C(6)-O(14) bonds having significantly different bond lengths, 1.412(1) and 1.438(1) Å respectively. The packing of the molecules in the crystal is rather loose, with no intermolecular H...H distances less than 2.4 Å.

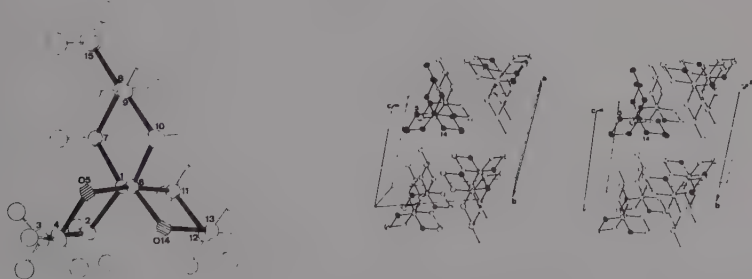


Fig. 1.  $C_{13}H_{22}O_2$ : molecular structure (left) and a stereoscopic view of the crystal packing (right).

DIBENZ[b,f]OXEPIN  
 $C_{14}H_{10}O$

J.A.G. DRAKE and D.W. JONES, 1982. *Acta Cryst.*, **B38**, 200-203.



Orthorhombic, Pnam,  $a = 8.236$ ,  $b = 6.115$ ,  $c = 19.667$  Å,  $D_m = 1.30$ ,  $Z = 4$ . Cu radiation,  $R = 0.115$  for 776 reflexions.

The molecules (Fig. 1), located on mirror planes passing through the O atom and the midpoint of the 1.327(6) Å C(10)-C(11) ethylenic bond, have almost planar benzene rings mutually inclined at a comparatively small dihedral angle of 134°. The heterocyclic ring, in boat conformation, has a C-O-C bond angle of 115.7° and C-O bonds of 1.390 Å.

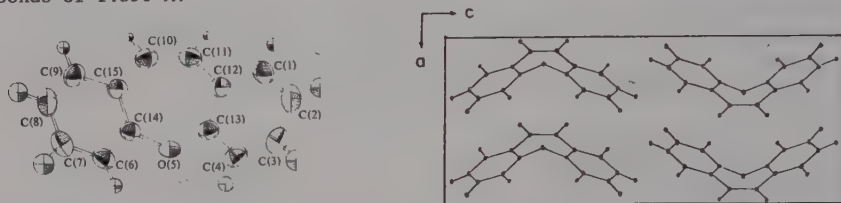


Fig. 1.  $C_{14}H_{10}O$ : a perspective view of the molecule and a view of the molecular packing along b.

#### BENZOCANTHARIDIN

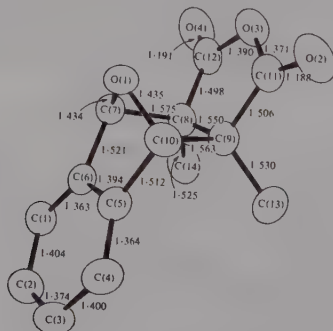
$C_{14}H_{12}O_4$



E.O. SCHLEMPER, T. SHINMYOZU and J.P. McCORMICK, 1982. *Acta Cryst.*, B38, 2981-2983.

Monoclinic,  $P2_1$ ,  $a = 7.511$ ,  $b = 7.297$ ,  $c = 11.084$  Å,  $\beta = 104.78^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.034$  for 1025 reflexions.

The structure of benzocantharidin (Fig. 1) is nearly identical to the parent cantharidin molecule (1) with some distortions due to the added fused aromatic ring. In particular the internal angles in the saturated six-membered rings, at the fusion with the aromatic ring, increase because of the shortening of the common C-C bond. Whereas the parent compound has crystallographic mirror symmetry, the present compound has only approximate mirror symmetry because of the crystallographic environment. Bond lengths and angles are normal. The crystal structure consists of discrete molecules with only van der Waals forces between them.



Orthorhombic,  $Pbca$ ,  $a = 31.250$ ,  $b = 7.742$ ,  $c = 11.277$  Å,  $Z = 8$ . Cu radiation,  $R = 0.040$  for 2024 reflexions.

The molecule consists of pyridine and furan rings joined by  $-\text{CONHN}=\text{CMe}-\text{CH}=\text{CH}(\text{C})$  chains (Fig. 1). The rings are planar within experimental error, and the dihedral angle between them is  $20^\circ$ . The chain is almost planar, the largest torsion angle being  $15^\circ$  about bond  $\text{N}(2)-\text{N}(3)$ . The bond lengths in the chain indicate extensive conjugation:  $\text{C}(6)-\text{N}(2)$  1.352,  $\text{N}(2)-\text{N}(3)$  1.387,  $\text{C}(7)-\text{N}(3)$  1.289,  $\text{C}(7)-\text{C}(9)$  1.458, and  $\text{C}(9)-\text{C}(10)$  1.331 Å. The water molecules are hydrogen-bonded to the molecule  $\text{N}(\text{pyridine})\cdots\text{O}$  2.89 Å and  $\text{N}(\text{hydrazine})\cdots\text{O}$  2.96 Å.

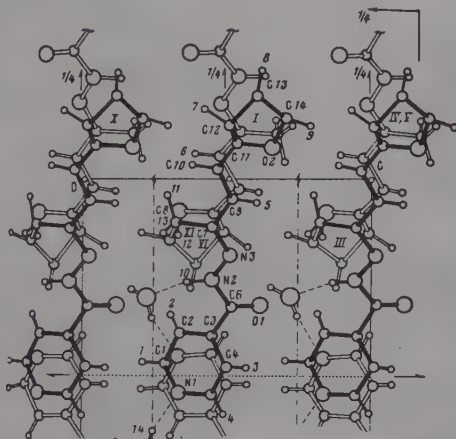


Fig. 1. Part of projection of structure of  $\text{C}_{14}\text{H}_{13}\text{N}_3\text{O}_2 \cdot \text{H}_2\text{O}$  on the  $ac$  plane.

1,2,3,4,5,8-HEXAHYDRO-endo-1,4-exo-5,8-DIMETHANONAPHTHALENE-4a,8a-DICARBOXYLIC ANHYDRIDE

$\text{C}_{14}\text{H}_{14}\text{O}_3$  (I)

DECAHYDRO-2,3-exo-EPOXY-1,4-endo-5,8-exo-DIMETHANONAPHTHALENE-4a,8a-DICARBOXYLIC ANHYDRIDE

$\text{C}_{14}\text{H}_{14}\text{O}_4$  (II)

1,2,3,4,4a,5,8,8a-OCTAHYDRO-5,8-exo-OXA-1,4-exo-METHANONAPHTHALENE-4a,8a-DICARBOXYLIC ANHYDRIDE

$\text{C}_{13}\text{H}_{12}\text{O}_4$  (III)

1,2,3,4,4a,5,8,8a-OCTAHYDRO-5,8-endo-OXA-1,4-exo-METHANONAPHTHALENE-4a,8a-DICARBOXYLIC ANHYDRIDE

$\text{C}_{13}\text{H}_{12}\text{O}_4$  (IV)

P.D. BARTLETT, G.L. COMBS, Jr., A.-X.T. LE, W.H. WATSON, J. GALLOY and M. KIMURA, 1982. *J. Am. Chem. Soc.*, **104**, 3131-3138.

I. Triclinic,  $P\bar{1}$ ,  $a = 7.164$ ,  $b = 8.289$ ,  $c = 10.145$  Å,  $\alpha = 96.30$ ,  $\beta = 92.22$ ,  $\gamma = 111.90^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.084$  for 1291 reflexions.

II. Monoclinic,  $P2_1/a$ ,  $a = 14.578$ ,  $b = 12.186$ ,  $c = 14.599$  Å,  $\beta = 119.22^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.088$  for 2114 reflexions.

III. Monoclinic,  $P2_1/c$ ,  $a = 15.982$ ,  $b = 7.564$ ,  $c = 12.084$  Å,  $\beta = 134.44^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.087$  for 1124 reflexions.

IV. Monoclinic,  $P2_1/c$ ,  $a = 11.472$ ,  $b = 7.635$ ,  $c = 12.260$  Å,  $\beta = 96.72^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.035$  for 961 reflexions.

The structures of I-IV are shown in Fig. 1. Bond lengths ( $\sigma = 0.004$ - $0.010$  Å) and angles ( $\sigma = 0.2$ - $1.0^\circ$ ) are normal.

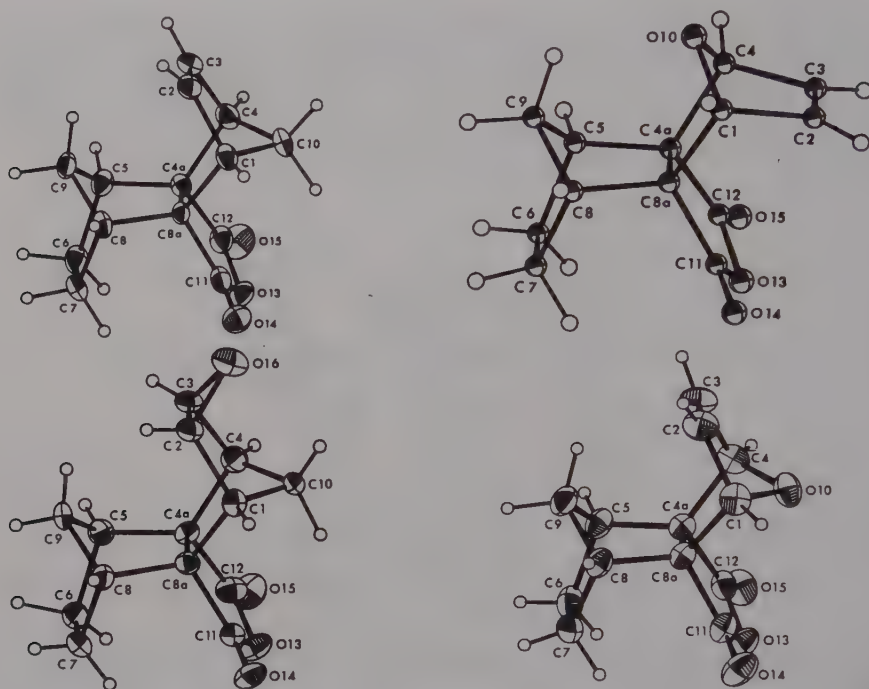


Fig. 1. The molecular structures of  $C_{14}H_{14}O_3$  (top left),  $C_{14}H_{14}O_4$  (bottom left),  $C_{13}H_{12}O_4$  [III] (top right), and  $C_{13}H_{12}O_4$  [IV] (bottom right).

1-(2',4'-DINITROPHENOXY)-9-OXABICYCLO[3.3.1]NONANE  
 $C_{14}H_{16}N_2O_6$

P.G. JONES, G.M. SHELDRICK, A.J. KIRBY, C.M. EVANS, R. GLENN and J. STEGEMANN, 1982. Z. Krist., 160, 45-51.

Orthorhombic,  $Pbca$ ,  $a = 10.293$ ,  $b = 22.665$ ,  $c = 12.218$  Å,  $Z = 8$ . Mo radiation,  $R = 0.065$  for 1600 reflexions.

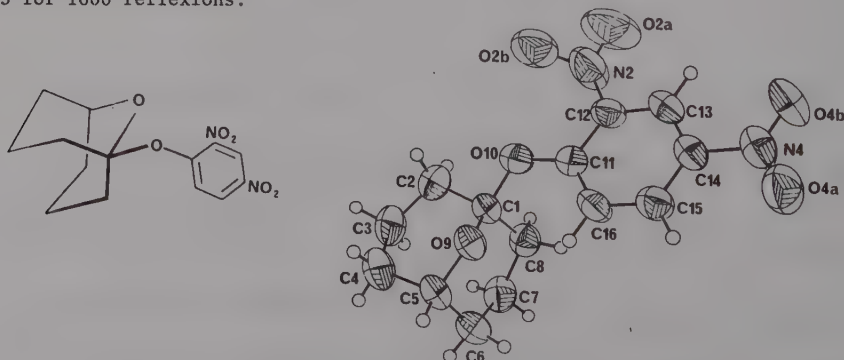


Fig. 1. A view of  $C_{14}H_{16}N_2O_6$ .

The compound shows an extraordinary combination of bond lengths at the acetal centre C(1) (Fig. 1). The C(1)-O(10) bond (1.448(5) Å) is very long for a C-O single bond but the endocyclic C(1)-O(9) bond (1.411(5) Å) is of normal length.

DIMETHYL 2,3-DIHYDRO-2,3-DIHYDROXY-4,7-DIMETHYLBENZO[b]FURAN-2,3-DICARBOXYLATE  
 $C_{14}H_{16}O_7$

F. THEOBALD and J. MORVAN, 1982. Z. Krist., **160**, 1-8.

Monoclinic,  $P2_1/n$ ,  $a = 15.376$ ,  $b = 7.397$ ,  $c = 12.598$  Å,  $\beta = 104.91^\circ$ ,  $D_m = 1.40$ ,  $Z = 4$ .  
 Cu radiation,  $R = 0.083$  for 2309 reflexions.

The analysis establishes that the compound has the cis-configuration of the methyl carboxylate groups (Fig. 1).

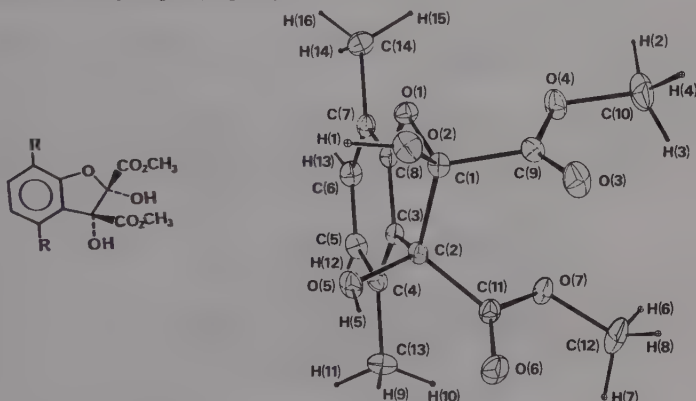


Fig. 1. The  $C_{14}H_{16}O_7$  cis-isomer ( $R = CH_3$ ).

1,4,7,10,13,16-HEXAOXACYCLOOCTADECANE METHYLAMMONIUM TRIFLUOROMETHANESULFONATE  
 $C_{14}H_{30}F_3NO_9S$   $C_{12}H_{24}O_6 \cdot CH_6N^+ \cdot CF_3O_3S^-$

A.L. SPEK, G. ROELOFSEN, J.G. NOLTES and A.H. ALBERTS, 1982. Cryst. Struct. Comm., **11**, 1863-1868.

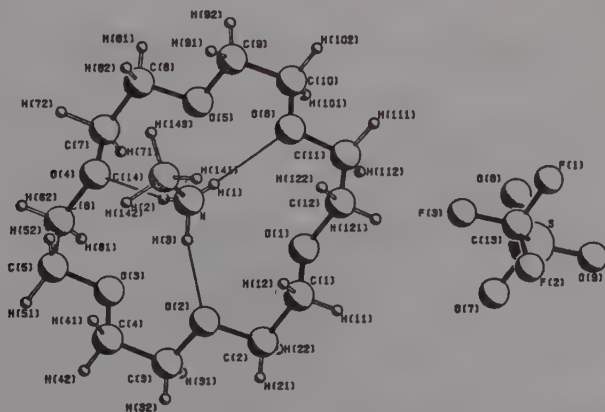


Fig. 1. A view of  $C_{12}H_{24}O_6 \cdot CH_6N^+ \cdot CF_3O_3S^-$ .



Monoclinic,  $P2_1/c$ ,  $a = 4.074$ ,  $b = 22.735$ ,  $c = 13.374$  Å,  $\beta = 96.08^\circ$ ,  $D_m = 1.61$ ,  $Z = 4$ . Mo radiation,  $R = 0.056$  for 1500 reflexions.

In the molecule (Fig. 1) the phenyl ring at C(2) is rotated  $45.9^\circ$  with respect to the remainder of the molecule because of the steric interaction between the Br atom at C(3) and the adjacent H atom on the phenyl ring even though the molecule has a conjugated bond system and could have been coplanar.

cis-3-BROMO-2,3-DIHYDRO-2-PHENYL-4H-1-BENZOPYRAN-4-ONE (3-BROMOFLAVANONE)  
 $C_{15}H_{11}BrO_2$

J.S. CANTRELL and G.S. HOCKSTEIN, 1982. Acta Cryst., B38, 981-983.

Monoclinic,  $P2_1/n$ ,  $a = 9.472$ ,  $b = 15.035$ ,  $c = 9.038$  Å,  $\beta = 95.70^\circ$ ,  $D_m = 1.58$ ,  $Z = 4$ . Mo radiation,  $R = 0.073$  for 1637 reflexions.

In the molecule (Fig. 1) the phenyl ring is rotated  $42.4^\circ$  with respect to the benzo ring, and the  $\gamma$ -pyrone ring is in the 1,2-diplanar conformation with the vicinal H atom in the cis configuration.

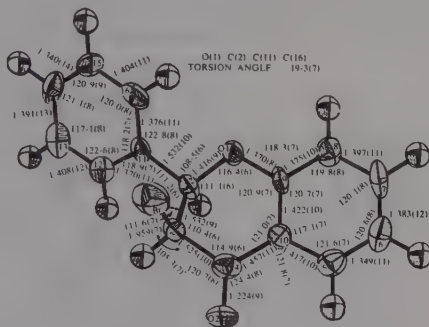


Fig. 1. 3-bromoflavanone: bond lengths and angles.

CONOCARPIN DIBROMOQUINOLIDE

$C_{15}H_{12}Br_2O_8$  (I)

CONOCARPIN DIBROMOCHROMANOL

$C_{15}H_{12}Br_2O_8$  (II)

J.C.A. BOYENS, M.U. NAUDE and G.W. PEROLD, 1982. S. Afr. J. Chem., 35, 157-160.

I. Monoclinic,  $P2_1$ ,  $a = 10.113$ ,  $b = 6.698$ ,  $c = 12.744$  Å,  $\beta = 97.08^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.25$  for 1316 reflexions.

II. Monoclinic,  $P2_1$ ,  $a = 10.683$ ,  $b = 5.738$ ,  $c = 14.076$  Å,  $\beta = 108.14^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.12$  for 1221 reflexions.

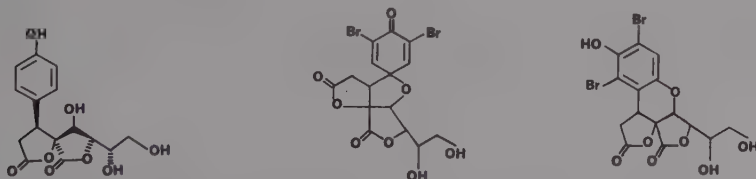


Fig. 1. The structures of conocarpin (left) and derivatives I (centre) and II (right).



The analyses establish the structures and absolute stereochemistry of conocarpin (Fig. 1). The analysis of I shows that there is probably disordered solvent in the lattice.

3,4-DIHYDRO-8-HYDROXY-3-(4-HYDROXYPHENYL)ISOCOUMARIN (HYDRANGENOL)



H.W. SCHMALLE, O.H. JARCHOW, B.M. HAUSEN and K.-H. SCHULZ, 1982. *Acta Cryst.*, **B38**, 2938-2941.

Monoclinic,  $P2_1$ ,  $a = 5.600$ ,  $b = 5.185$ ,  $c = 20.827$  Å,  $\beta = 97.52^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.037$  for 786 reflexions.

In the molecule (Fig. 1) the 4-hydroxyphenyl ring C is equatorially substituted at C(3) of the slightly distorted sofa form of the lactone ring B. The conformation of the molecule is characterized by two planes of ring A and ring C which are approximately perpendicular ( $78.8^\circ$ ) to each other. The hydroxyl H at O(8) is hydrogen bonded to the C(1) carbonyl group with distances O(8)-H(8) 0.96(6) and O(1)...H(8) 1.82(12) Å, the angle O(8)-H(8)...O(1) being  $138(4)^\circ$ . Intermolecular hydrogen bonds of the type O-H...O form endless chains with alternate molecules along b.

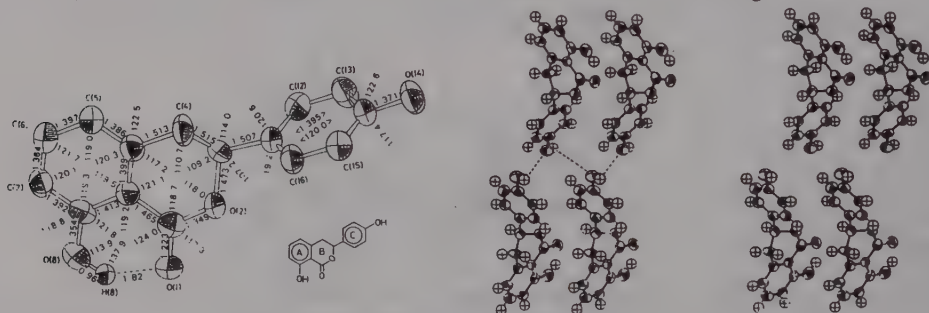


Fig. 1.  $\text{C}_{15}\text{H}_{12}\text{O}_4$ : bond lengths and angles in the molecule and a stereodrawing of the bc projection of the crystal structure.

2'-HYDROXY-6-METHOXY-4,5'-DIMETHYLSPIRO(BENZOFURAN-2(3H),1'-CYCLOPENT-4'-ENE)-3(2H),3'-DIONE



T. SALA, M.V. SARGENT, B.W. SKELTON and A.H. WHITE, 1982. *Aust. J. Chem.*, **35**, 1065-1070.

Triclinic,  $P\bar{1}$ ,  $a = 12.436$ ,  $b = 7.622$ ,  $c = 6.986$  Å,  $\alpha = 89.88^\circ$ ,  $\beta = 82.88^\circ$ ,  $\gamma = 81.52^\circ$ ,  $D_m = 1.40$ ,  $Z = 2$ . Mo radiation,  $R = 0.043$  for 1803 reflexions.

The five-membered ring fused to the benzene ring (Fig. 1) is nearly planar ( $\sigma$  0.017 Å) and the dihedral angle between the two ring planes is  $2.5^\circ$ . The spiro-fused five-membered ring conformation approximates an envelope with C(2') at the flap. Bond lengths and angles have expected values. Molecules are joined together by a strong hydrogen bond (H(2'A)...O(3') 1.96(3) Å).

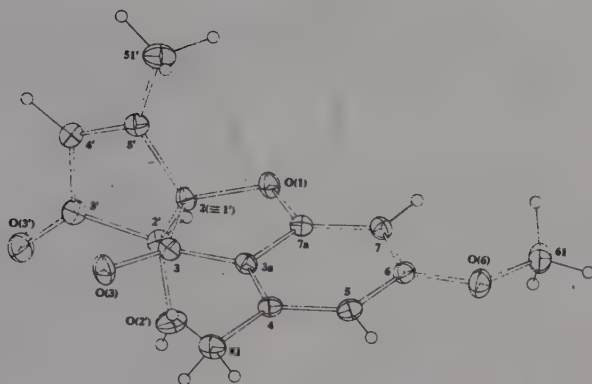


Fig. 1. Projection of  $C_{15}H_{14}O_5$  normal to the aromatic ring plane.

ETHYL  $\alpha$ -PHENYLHYDRAZONO-2-FURANPROPIONATE



PUTTARAJA, D.S.S. GOWDA, E.J. GABE and Y. LE PAGE, 1982. *Acta Cryst.*, **B38**, 692-694.

Orthorhombic,  $Pca2_1$ ,  $a = 11.115$ ,  $b = 8.170$ ,  $c = 15.341$  Å,  $D_m = 1.290$ ,  $Z = 4$ . Cu radiation,  $R = 0.041$  for 1033 reflexions.

In the molecule (Fig. 1) the  $N=N-C-O$  fragment is almost planar with bond angles typical of  $sp^2$  hybridisation. The  $C-N-N-C$  torsion angle is  $-178.4^\circ$  and the  $N-N$  bond length is  $1.333$  Å. Molecules are linked along  $a$  by  $N-H \cdots O$  hydrogen bonds.

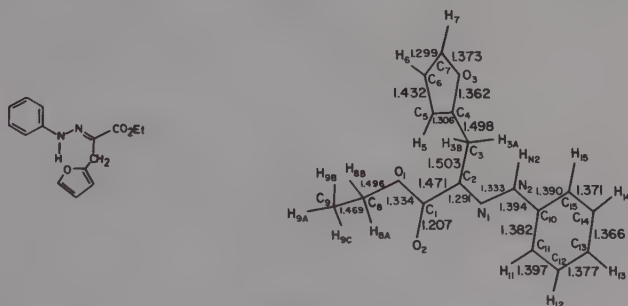


Fig. 1. The  $C_{15}H_{16}N_2O_3$  molecule and bond lengths.

7a-BROMO-2,2a,2b,7,7a,7b-HEXAHYDRO-7-OXO-1H-BENZO[f]CYCLOPROP[cd]INDENE-1,2-DICARBOXYLIC ANHYDRIDE



A. HASSNER, D. MIDDLEMISS, J. MURRAY-RUST and P. MURRAY-RUST, 1982. *Tetrahedron*, **38**, 2539-2546.

Monoclinic,  $P2_1/c$ ,  $a = 10.09$ ,  $b = 9.91$ ,  $c = 13.28$  Å,  $\beta = 110.85^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.070$  for 1559 reflexions.

The structure is determined to be as shown in Fig. 1.

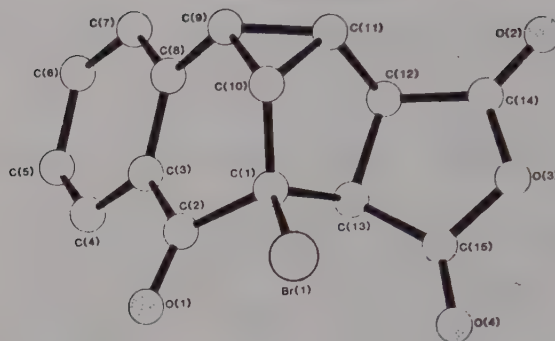


Fig. 1. The structure of  $C_{15}H_{19}BrO_4$ .

1,4-DIOXASPIRO[4.5]DEC-8-YL p-TOLUENESULPHONATE  
 $C_{15}H_{20}O_5S$

G. BOCELLI, M.-F. GRENIER-LOUSTALOT and P. IRATCABAL, 1982. *Acta Cryst.*, **B38**, 2068-2070

Triclinic,  $P\bar{1}$ ,  $a = 11.593$ ,  $b = 11.258$ ,  $c = 6.808$  Å,  $\alpha = 104.6$ ,  $\beta = 94.7$ ,  $\gamma = 112.8^\circ$ ,  $Z = 2$ .  $^{\circ}\text{Cu}$  radiation,  $R = 0.064$  for 2585 reflexions.

The mean planes of the cyclohexane and dioxolane rings (which have the chair and envelope conformations respectively) are inclined at  $86.0(4)^\circ$ ; C(7) is displaced 0.42 Å from the mean ring plane (Fig. 1). The O(1)-S-O(2) plane is inclined at  $87.0(3)^\circ$  to C(9)-S-O(3) and the O(1)-S-O(2), O(1)-S-O(3), O(3)-S-C(9) angles are  $119.2$ ,  $102.9$  and  $104.4^\circ$ . There are no short intermolecular contacts.

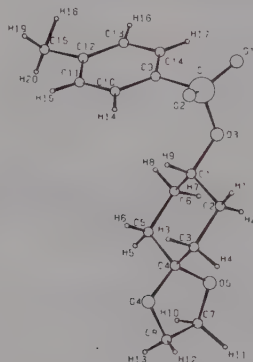
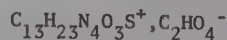


Fig. 1. The  $C_{15}H_{20}O_5S$  molecule.

RANITIDINE HYDROGEN OXALATE  
 $C_{15}H_{24}N_4O_7S$



B. KOJIC-PRODIC, Z. RUZIC-TOROS and R. TOSO, 1982. *Acta Cryst.*, **B38**, 1837-1840.

Monoclinic,  $P2_1/c$ ,  $a = 13.642$ ,  $b = 5.531$ ,  $c = 26.018$  Å,  $\beta = 103.51^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.048$  for 1676 reflexions.

The protonated ranitidine molecule exhibits electron-delocalisation in the 2-ethylamino-2-methylamino-1-nitroethylene residue (C(1)-C(2) 1.433(8), C(1)-N(3) 1.321(7), C(2)-N(5) 1.353(7), C(1)-N(8) 1.315(6) Å) and is folded head-to-tail

(Fig. 1) with an intramolecular hydrogen bond (N(3)...O(6) 2.598(6) Å). Each planar hydrogen oxalate ion is hydrogen-bonded to two ranitidine ions (N...O 2.797(6) and 2.921(6) Å) forming chains along c. The hydrogen oxalate ions are connected into chains along b by short hydrogen bonds (O...O 2.461(5) Å) giving layers of anions and cations in the bc plane.

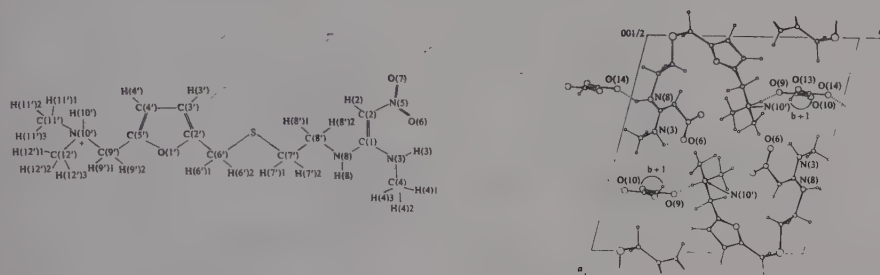


Fig. 1. Molecular skeleton, packing and hydrogen bonding in ranitidine hydrogen oxalate.

(-)-6-BROMOCRYPTOSTROBIN

$C_{16}H_{13}BrO_4$

L.T. BYRNE, J.R. CANNON, D.H. GAWAD, B.S. JOSHI, B.W. SKELTON, R.F. TOIA and A.H. WHITE, 1982. *Aust. J. Chem.*, **35**, 1851-1858.

Tetragonal,  $P4_32_12$ ,  $a = 7.849$ ,  $c = 46.83$  Å,  $D_m = 1.59$ ,  $Z = 8$ . Mo radiation,  $R = 0.050$  for 810 reflexions.

The structure was established as that shown in Fig. 1. Bond lengths are generally as expected although C(8)-C(7), C(81) (1.46(1), 1.62(1) Å) are unusually long. Within the fused aromatic ring the angle at C(6) (122.9(9)°) and the angles at C(2) and C(3) in the heterocyclic ring (111.1(7), 111.3(8)°) are larger than normal.

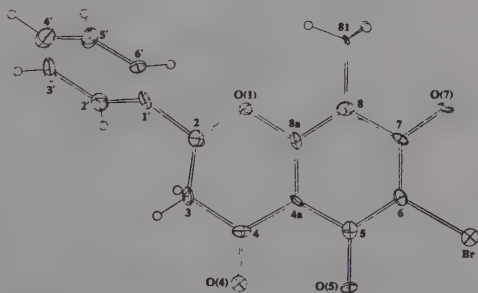


Fig. 1. Projection of (-)-6-bromocryptostrobin onto the fused aromatic ring plane (phenolic hydrogen atoms are not shown).

14-OXO-1,2-DEHYDROCACALOL METHYL ETHER

$C_{16}H_{16}O_3$

L.T. GELBAUM, D. VAN DERVEER and L.H. ZALKOW, 1982. *Cryst. Struct. Comm.*, **11**, 1765-1769.

Orthorhombic,  $P2_12_12_1$ ,  $a = 8.687$ ,  $b = 5.229$ ,  $c = 27.89$  Å,  $D_m = 1.30$ ,  $Z = 4$ . Mo radiation,  $R = 0.075$  for 975 reflexions.

The structure (Fig. 1) determined by this analysis confirms the structure established by a total synthesis.

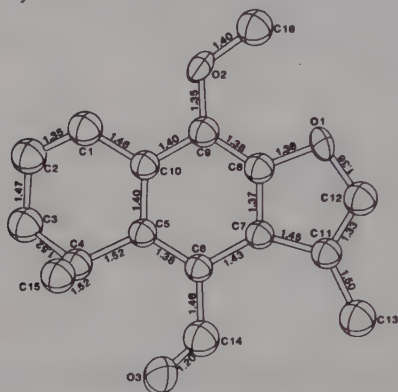


Fig. 1. The  $C_{16}H_{16}O_3$  molecule and bond lengths ( $\sigma$  0.006–0.008 Å).

4-METHOXY-5-METHYL-5-PHENYLTHIOCYCLOHEX-3-ENE-1,2-DICARBOXYLIC ANHYDRIDE  
 $C_{16}H_{16}O_4S$

A.P. KOZIKOWSKI and E. HUIE, 1982. J. Am. Chem. Soc., 104, 2059–2060.

Orthorhombic,  $P2_12_12_1$ ,  $a = 6.512$ ,  $b = 8.332$ ,  $c = 27.603$  Å,  $Z = 4$ . Cu radiation,  $R = 0.057$  for 1137 reflexions.

The structure is shown in Fig. 1. Bond lengths and angles are normal.

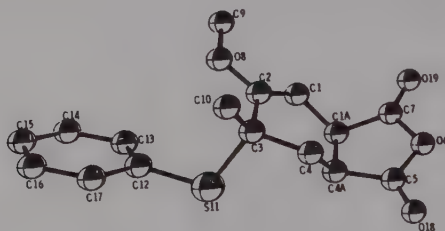


Fig. 1. The structure of  $C_{16}H_{16}O_4S$ .

SPIRO[CYCLOHEXANE-1,2'-INDAN]-1,4-DIONE CYCLIC 4-(ETHYLENE KETAL)  
 $C_{16}H_{18}O_3$

K.R. STEWART and J. BORDNER, 1982. Cryst. Struct. Comm., 11, 125–128.

Monoclinic,  $P2_1/c$ ,  $a = 10.053$ ,  $b = 8.383$ ,  $c = 19.489$  Å,  $\beta = 124.42^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.061$  for 1324 reflexions.

The conformation of the cyclohexane ring (Fig. 1) is a normal chair form in which the carbonyl of the spiro five-membered ring is in the equatorial position.

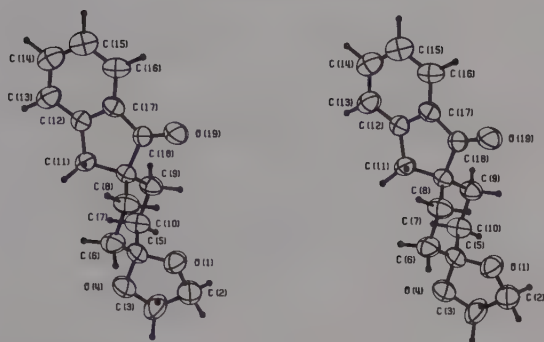


Fig. 1. A stereoview of  $C_{16}H_{18}O_3$ .

N-(6-endo-METHOXY-3-OXO-2-OXABICYCLO[3.2.1]OCT-8-YL)-N-METHYL-p-TOLUENESULPHONAMIDE  
 $C_{16}H_{21}NO_5S$

P. MURRAY-RUST, R.C. GLEN and R.F. NEWTON, 1982. *Acta Cryst.*, **B38**, 2696-2698.

Monoclinic,  $P2_1/c$ ,  $a = 6.46$ ,  $b = 11.77$ ,  $c = 22.62$  Å,  $\beta = 101.97^\circ$ ,  $D_m = 1.35$ ,  $Z = 4$ .  
 Mo radiation,  $R = 0.057$  for 1655 reflexions.

In the molecule (Fig. 1) the five-membered ring has a strongly puckered envelope conformation with an approximate mirror plane through C(8). The conformation about the C(6)-C(7) bond is almost eclipsed and steric hindrance appears to prevent any interaction between the methoxy and lactone groups. The lactone ring has a sofa conformation with C(1), O(2), C(3), C(4) and C(5) almost coplanar. The crystal structure consists of discrete molecules with no unusually short intermolecular contacts.

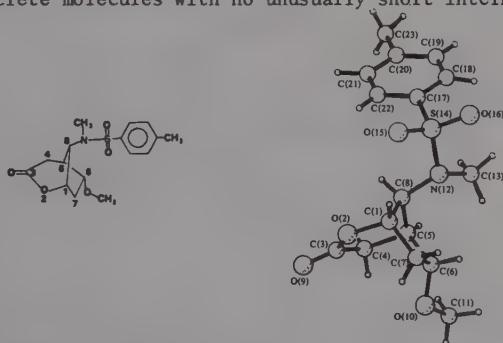


Fig. 1.  $C_{16}H_{21}NO_5S$ : perspective view of the molecule; the methyl group at C(23) is disordered and only the most important conformation is shown.

2,2',4,4',6,6'-HEXAMETHYL-BIS-4,4'-(4H)-PYRANE  
 $C_{16}H_{22}O_2$

Z. DAUTER, S.A. CHAUDHURY and M.A. HAMID, 1982. *Cryst. Struct. Comm.*, **11**, 1005-1008.

Monoclinic,  $P2_1/c$ ,  $a = 7.838$ ,  $b = 11.531$ ,  $c = 8.846$  Å,  $\beta = 113.56^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.11$  for 655 reflexions.

The molecule (Fig. 1) lies on an inversion centre and has approximate  $2/m$  symmetry. The pyran ring is planar to within 0.04 Å.



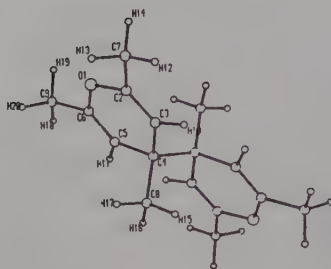


Fig. 1. A view of  $C_{16}H_{22}O_2$ .

2,3,11,12-TETRACARBOXYLATO-18-CROWN-6 OXONIUM CHLORIDE

$C_{16}H_{27}ClO_{15}$

$C_{16}H_{24}O_{14} \cdot (H_3O)^+ Cl^-$

J.-P. BEHR, P. DUMAS and D. MORAS, 1982. *J. Am. Chem. Soc.*, **104**, 4540-4543.

Orthorhombic,  $P2_12_12_1$ ,  $a = 10.526$ ,  $b = 14.325$ ,  $c = 15.234$  Å,  $Z = 4$ . Cu radiation,  $R = 0.048$  for 1957 reflexions (absolute configuration).

Two carboxylic acid groups extend on both sides of the pseudoplanar macrocycle. The  $H_3O^+$  cation is anchored in the centre of the cavity by three  $OH \cdots O$  hydrogen bonds (2.67, 2.73 and 2.74 Å) as shown in Fig. 1 whereas the  $Cl^-$  counterion is H-bonded to three carboxylic acid groups 5.5 Å away from the cation. The  $H_3O^+$  cation has pyramidal geometry with  $H-O-H$  95(5), 102(5), and 120(5)°.

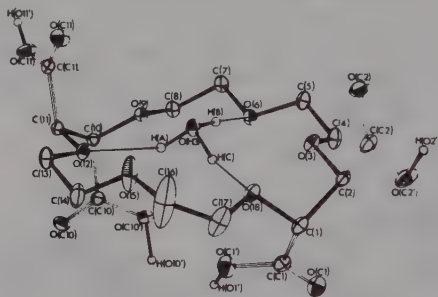


Fig. 1. The  $[C_{16}H_{24}O_{14} \cdot H_3O^+]$  complex cation.

1,4,10,13-TETRAOXA-7,16-DIAZACYCLOOCTADECANE-7,16-N,N'-DIACETATOCOPPER(II)

$C_{16}H_{28}CuN_2O_8$

P. GLUZINSKI, J.W. KRAJEWSKI, Z. URBANCZYK-LIPKOWSKA, J. BLEIDELIS and A. MISHNYOV, 1982. *Cryst. Struct. Comm.*, **11**, 1589-1592.

Triclinic,  $P\bar{1}$ ,  $a = 7.010$ ,  $b = 7.621$ ,  $c = 9.074$  Å,  $\alpha = 81.33$ ,  $\beta = 70.54$ ,  $\gamma = 86.35^\circ$ ,  $Z = 1$ . Cu radiation,  $R = 0.047$  for 1391 reflexions.

The Cu atom (Fig. 1) lies on a crystallographic inversion centre, with Cu-N 2.055(4), Cu-O(7) 2.645(4), Cu-O(13) 1.943(3) Å.

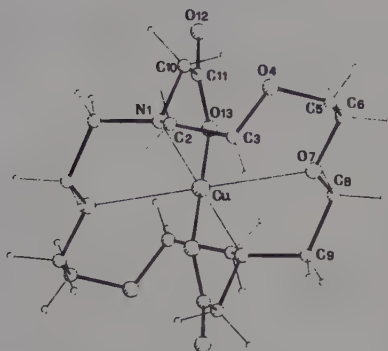
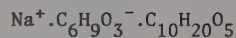
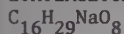


Fig. 1. The  $C_{16}H_{28}CuN_2O_8$  molecule.

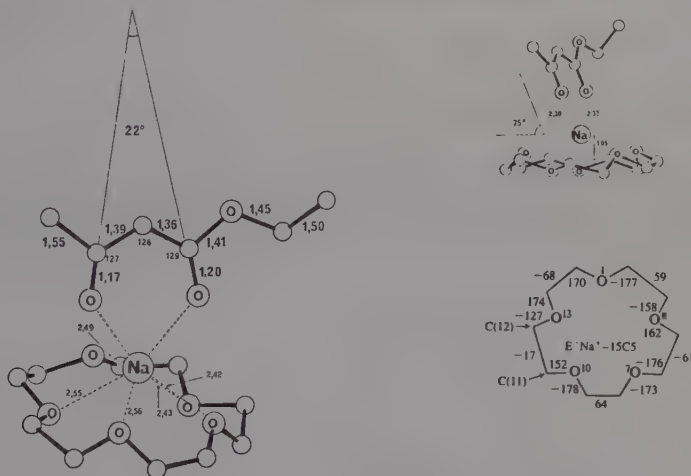
ETHYLACETOACETATE SODIUM ENOLATE 15-CROWN-5



C. CAMBILLAU, G. BRAM, J. CORSET and C. RICHE, 1982. *Canad. J. Chem.*, **60**, 2554-2565.

Orthorhombic,  $P2_12_12_1$ ,  $a = 9.150$ ,  $b = 13.127$ ,  $c = 16.566$  Å,  $Z = 4$ . Cu radiation,  $R = 0.086$  for 1038 reflexions.

The  $Na^+$  ion (Fig. 1) is  $1.05$  Å out of the mean plane of the crown; principal dimensions are also shown in Fig. 1.



molecules and one disordered aniline. Two nitroaniline molecules, related by a centre of inversion, are inclined with dihedral angles  $66(1)^\circ$  (for A) and  $53(1)^\circ$  (for B) (Fig. 1) above and below the planes formed by the six O atoms of the crown molecules (coplanar within  $\pm 0.026 \text{ \AA}$ ). Four strong N-H...O hydrogen bonds ( $2.893(5)$ - $3.200(5) \text{ \AA}$ ) link the guest and host molecules.

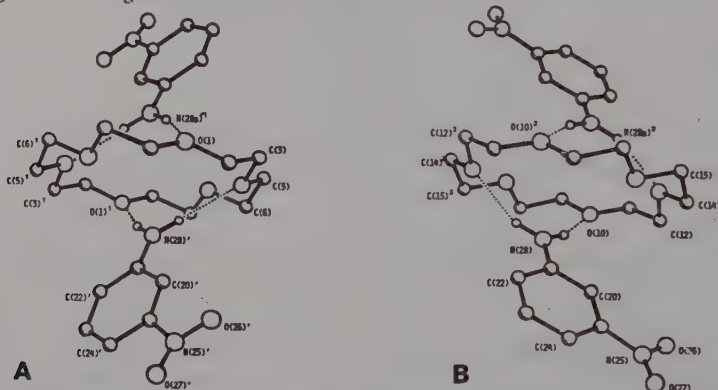
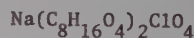
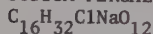


Fig. 1. Perspective views of the independent  $C_{12}H_{24}O_6, C_6H_6N_2O_2$  adducts. The strong N-H...O hydrogen bonds are shown by dotted lines.

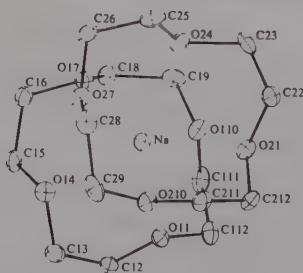
# SODIUM PERCHLORATE 1,4,7,10-TETRAOXACYCLODODECANE (1:2)

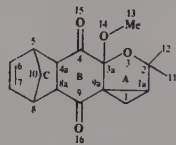


E. MASON and H.A. EICK, 1982. *Acta Cryst.*, B38, 1821-1823.

Monoclinic,  $P2_1/a$ ,  $a = 15.420$ ,  $b = 15.245$ ,  $c = 9.650 \text{ \AA}$ ,  $\beta = 92.13^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.07$  for 1401 reflexions.

The  $Na^+$  ion is coordinated by eight oxygen atoms from two 12-crown-4 rings in a slightly distorted, rectangular, antiprismatic arrangement (Fig. 1) with Na...O distances in the range  $2.474(8)$  to  $2.543(8) \text{ \AA}$ .  $Na^+$  is  $1.53(1) \text{ \AA}$  from each of the rings which are planar to within  $\pm 0.01 \text{ \AA}$ . The disordered perchlorate groups form layers between sodium-dicrown layers with no significant interaction between them.



$$\text{C}_{17}\text{H}_{20}\text{O}_4$$


Monoclinic,  $P2_1/c$ ,  $a = 7.348$ ,  $b = 22.695$ ,  $c = 9.386$  Å,  $\beta = 112.5^\circ$ ,  $D_m = 1.32$ ,  $Z = 4$ .  
Cu radiation,  $R = 0.051$  for 1598 reflexions.

Ring A (see above) is an envelope conformation with O(3) at the flap and the cis-fused ring B is a flattened boat twisted about C(3a), C(8a). The dihedral angle between planes C(3a), C(4), C(4a), O(15) and C(8a), C(9), C(9a), O(16) is 39.6°; that between C(3a), C(4), C(4a), C(8a), C(9), C(9a) and C(1a), C(2), O(3), C(3a), C(9a) is 75.9°. Rings B and C are cis-fused. Noteworthy bond lengths are: C(1)-C(1a) 1.475(4), C(1)-C(9a) 1.517(5), C(1a)-C(9a) 1.522(4), C(3a)-C(9a) 1.503(4), C(4a)-C(8a) 1.560(4), C(4a)-C(5) 1.567(5), C(8)-C(8a) 1.564(4) Å.

$$\text{C}_{17}\text{H}_{20}\text{O}_8$$

BOGATSKY, 1982. Acta Cryst., B38, 2955-2958.

Monoclinic,  $P2_1/b$ ,  $a = 10.798$ ,  $b = 9.057$ ,  $c = 17.585$  Å,  $\gamma = 93.02^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.048$  for 2254 reflexions.

Bond lengths and angles in the molecule (Fig. 1) are in good agreement with previously published values; the mean C-O and C-C distances (1.435(4) and 1.506(5) Å respectively) are characteristic of crown ethers. The 15-membered ring forms a distorted square. The three ester groups are in the trans form: two groups are almost perpendicular to the mean plane of the macrocycle and the third is displaced from it. The overall conformation is stabilized by both intra- and intermolecular C=O...C=O and C-H...O (ester or ether) interactions.

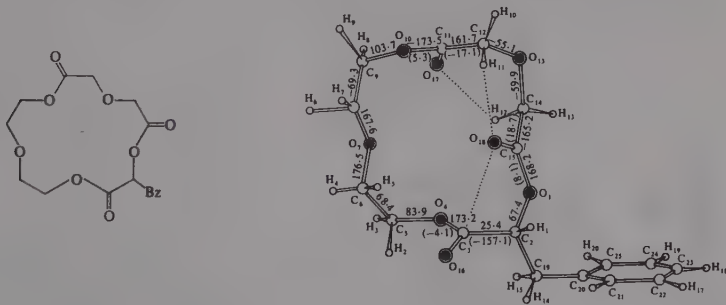


Fig. 1.  $C_{17}H_{20}O_8$ : a perspective view of the molecule showing the torsion angles.

DIMETHYL (1SR,9RS)-11,11-DIMETHYL-4-OXO-5-OXATRICYCLO[7.3.0.0<sup>3,7</sup>]DODEC-3-ENE-2,2-DICARBOXYLATE

$C_{17}H_{22}O_6$

R.K. BOECKMAN, Jr. and S.S. KO, 1982. *J. Am. Chem. Soc.*, **104**, 1033-1041.

Monoclinic,  $P2_1/c$ ,  $a = 15.370$ ,  $b = 9.399$ ,  $c = 12.056$  Å,  $\beta = 97.33^\circ$ ,  $D_m = 1.06$ ,  $Z = 4$ . Mo radiation,  $R = 0.058$  for 1510 reflexions.

The molecule (Fig. 1) contains a system of three fused rings. Bond lengths and angles are normal.

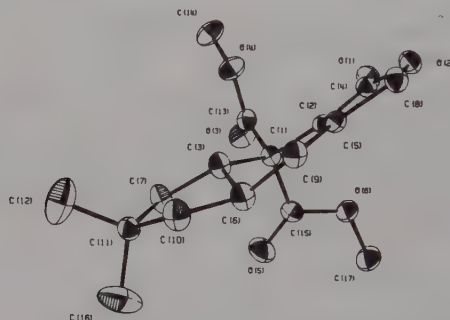


Fig. 1. The structure of  $C_{17}H_{22}O_6$ .

10 $\alpha$ -t-BUTYL-3,3 $\alpha$ ,4,5,6,6 $\alpha$ ,7,8,9,10-DECAHYDRO-7 $\alpha$ -METHYL-1H-NAPHTHO[1,8a-c]FURAN-1,8-DIONE

$C_{17}H_{26}O_3$

B. RUBIN, T. DANOFF and L. BROOKS, 1982. *Acta Cryst.*, **B38**, 1033-1035.

Orthorhombic,  $P2_12_12_1$ ,  $a = 11.598$ ,  $b = 9.382$ ,  $c = 13.985$  Å,  $D_m = 1.21$ ,  $Z = 4$ . Cu radiation,  $R = 0.03$  for 882 reflexions.

In the molecule (Fig. 1) the t-butyl group is axial. The bond angles about C(15) are  $106$ – $109^\circ$  and suggest no particular distortion, but the C(3)-C(15) bond is long ( $1.577$  Å). The bond angles in the furanone ring system suggest considerable strain, as is found in other similar systems.

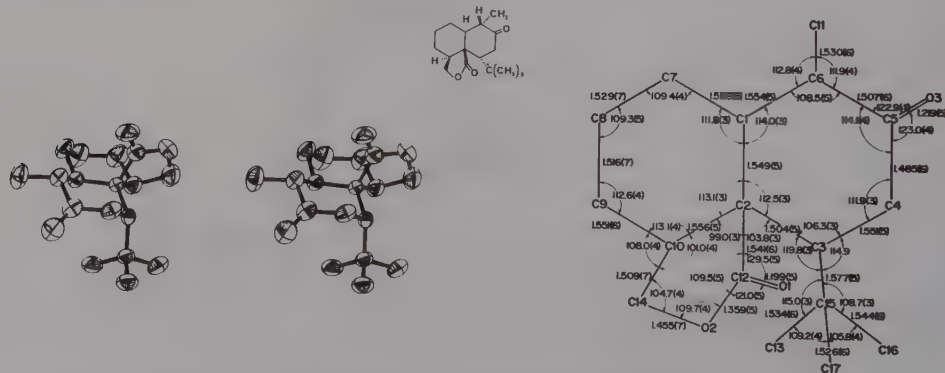


Fig. 1.  $C_{17}H_{26}O_3$ : a stereoscopic view of the molecule (left) and bond lengths and angles (right).

(3R) (4R) (6R) -6-METHYL-3-PHENYL-4-(p-CHLOROPHENYL)TETRAHYDROPYRAN-2-ONE  
 $C_{18}H_{17}ClO_2$

S. AXIOTIS, J. DREUX, M. PERRIN and J. ROYER, 1982. *Tetrahedron* **38**, 499-504.

Monoclinic,  $P2_1/a$ ,  $a = 13.687$ ,  $b = 10.707$ ,  $c = 10.723$  Å,  $\beta = 102.14^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.062$  for 3147 reflexions.

The six-membered ring (Fig. 1) has a slightly twisted boat conformation.

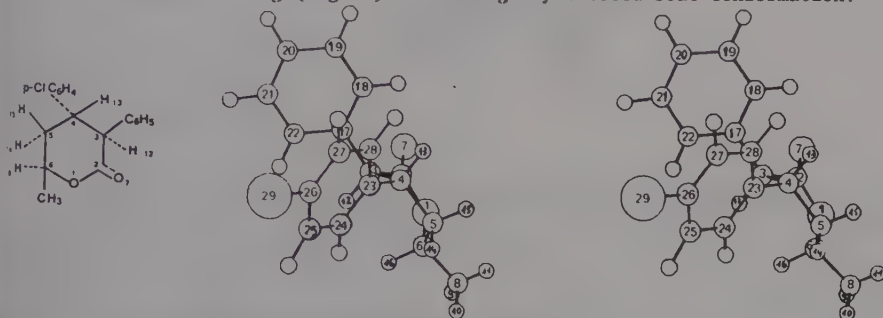


Fig. 1. The  $C_{18}H_{17}ClO_2$  molecule and a stereoview.

4-(2-NITROPHENYLTHIO)-12-CHLOROMETHYL-9-OXAPENTACYCLO[6.4.0.0.2<sup>5</sup>.0<sup>3</sup>.7.0<sup>6</sup>.1<sup>11</sup>]DODECANE  
 $C_{18}H_{18}ClNO_3S$

N.S. ZEFIROV, V.N. KIRIN, A.S. KOZ'MIN, K.A. POTEKHIN, A.I. YANOVSKY and Yu.T. STRUCHKOV, 1982. *Cryst. Struct. Comm.*, **11**, 721-726.

Triclinic,  $P\bar{1}$ ,  $a = 6.622$ ,  $b = 9.733$ ,  $c = 13.724$  Å,  $\alpha = 74.32^\circ$ ,  $\beta = 78.40^\circ$ ,  $\gamma = 76.88^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.063$  for 1442 reflexions.

The analysis provided unambiguous proof of the participation of ether ring oxygen acting as a nucleophile in the final step of addition with formation of a six-membered oxygen-containing ring in the compound (Fig. 1).

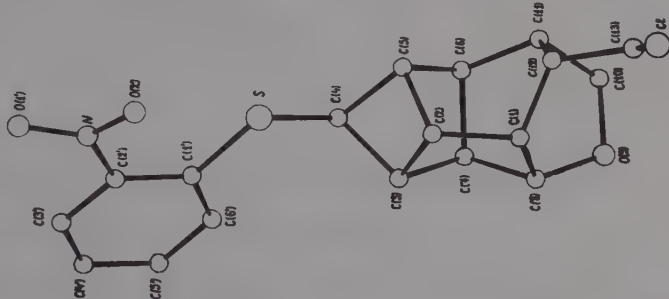


Fig. 1. A view of  $C_{18}H_{18}ClNO_3S$ .

(4aR,5aR,10bS)-10b-HYDROXY-5a-METHOXY-2,2-DIMETHYL-2H,5aH-OXIRENO[2,3]NAPHTHO[1,2-b]-PYRAN-6(10bH)-ONE ACETATE

$C_{18}H_{18}O_6$

R.M. WILSON, T.F. WALSH and R. WHITTLE, 1982. *J. Am. Chem. Soc.*, **104**, 4162-4166.

Orthorhombic,  $Pna2_1$ ,  $a = 17.261$ ,  $b = 8.680$ ,  $c = 11.087$  Å,  $D_m = 1.324$ ,  $Z = 4$ . Mo radiation,  $R = 0.066$  for 1834 reflexions.



The molecule was found to possess the structure shown in Fig. 1. No unusual structural features were noted.

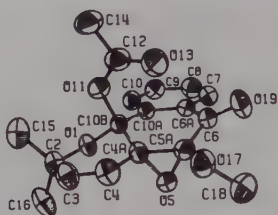


Fig. 1. The  $C_{18}H_{18}O_6$  molecule.

7-CYANO-5,6,6-TRIMETHYL-8,9-BENZO-3-OXATRICYCLO[5.4.0.0<sup>1,5</sup>]-8,10-UNDECADIENE  
 $C_{18}H_{19}NO$

J.J. McCULLOUGH, W.K. MacINNIS, C.J.L. LOCK and R. FAGGIANI, 1982. J. Am. Chem. Soc., 104, 4644-4658.

Monoclinic,  $P2_1/c$ ,  $a = 12.089$ ,  $b = 8.163$ ,  $c = 15.152$  Å,  $\beta = 105.27^\circ$ ,  $D_m = 1.20$ ,  $Z = 4$ . Mo radiation,  $R = 0.064$  for 2773 reflexions.

The structure of the tricyclic molecule is shown in Fig. 1. The olefinic C(10)-C(11) bond is short at 1.317(4) Å and is not conjugated with the aromatic system. The cyclobutano ring is planar with long (1.575(4) to 1.592(3) Å) C-C bonds. The tetrahydrofuran ring has an O-envelope conformation: C-O 1.427(3) and 1.425(4), C(1)-C(5) 1.586(3), C(1)-C(2) 1.521(4), C(4)-C(5) 1.529(4) Å.

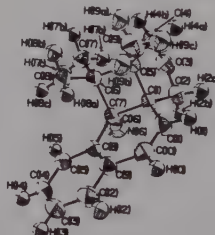
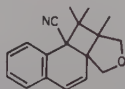


Fig. 1. The  $C_{18}H_{19}NO$  molecule.

ETHYL t-2,3-EPOXY-c-6-p-METHOXYBENZOYLOXY-1-METHYLCYCLOHEXANE-r-1-CARBOXYLATE  
 $C_{18}H_{22}O_6$

H.M. SIRAT, E.J. THOMAS and J.D. WALLIS, 1982. J. Chem. Soc. Perkin I, 2885-2896.

Triclinic,  $P\bar{1}$ ,  $a = 8.860$ ,  $b = 9.998$ ,  $c = 12.439$  Å,  $\alpha = 63.54$ ,  $\beta = 67.18$ ,  $\gamma = 68.65^\circ$ ,  $Z = 2$ . Radiation not stated,  $R = 0.056$  for 1604 reflexions.

The C(4)-C(5) bond in the cyclohexene epoxide unit (Fig. 1) is unusually short (1.505 Å). This bond shortening has not been observed in three other cyclohexene epoxides (1,2), a value of 1.549 Å being reported for 1-phenyl-4-t-butylcyclohex-1-ene epoxide (1) (2). Atoms O(4) and H(51) are arranged almost antiperiplanar about C(4)-C(5), the torsion angle O(4)-C(4)-C(5)-H(51) being  $174.6^\circ$ . There is also asymmetry in the bond angles between the C(4)-O(4) bond and the cyclohexane ring bonds to C(4) (O(4)-C(4)-C(3)  $104.2^\circ$  and O(4)-C(4)-C(5)  $109.9^\circ$ ). There is a short non-bonded distance between O(4) and the carbonyl carbon C(8) (2.57 Å). The epoxide bonds C(1)-O(1) 1.428 and C(2)-O(1) 1.432 Å are more similar to mean values for unfused epoxides than to the mean value in I.

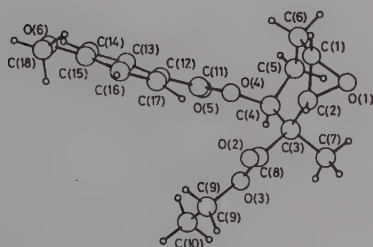


Fig. 1. Molecular structure of  $C_{18}H_{22}O_6$ .

1. Structure Reports, 38B, 334.
2. Ibid, 46B, 327.

(3S,7R,11E)-3,4,5,6,7,8,9,10-OCTAHYDRO-7,14,16-TRIHIDROXY-3-METHYL-1H-2-BENZOXACYCLO-TETRADECEN-1-ONE METHANOL SOLVATE ( $\alpha$ -ZEARALENOL)

$C_{18}H_{24}O_5 \cdot CH_4O$

W.H. WATSON, V. ZABEL, C.J. MIROCHA and S.V. PATHRE, 1982. Acta Cryst., B38, 1037-1040.

Monoclinic,  $P2_1$ ,  $a = 11.212$ ,  $b = 8.487$ ,  $c = 10.319$  Å,  $\beta = 101.55^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.078$  for 1783 reflexions.

The molecule (Fig. 1) consists of a 14-membered lactone ring fused ortho to a 1,3-dihydroxybenzene moiety. The phenyl ring and C(1), O(1), O(2), C(3), C(12), O(14) and O(16) form a planar system. The coplanarity of the phenyl ring and the conjugated carbonyl is further assisted by an internal hydrogen bond between O(1) and O(16) of 2.502 Å. The conformation is similar to that of 5-hydroxyzearealenone (1). A methanol of solvation is in a position to be hydrogen-bonded to three  $\alpha$ -zearealenol molecules.

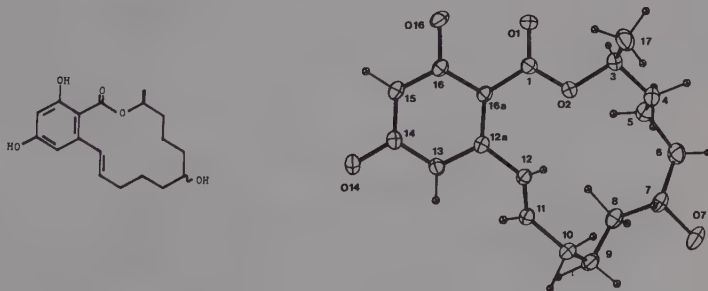


Fig. 1. A view of the  $\alpha$ -zearealenol molecule.

1. Structure Reports, 42B, 250.

2-METHYL-2-(2-NAPHTHYLOXY)-4H-1,3-BENZODIOXIN-4-ONE

$C_{19}H_{14}O_4$

J.-E. JØRGENSEN and A.B. HANSEN, 1982. Acta Cryst., B38, 991-993.

Monoclinic,  $P2_1/c$ ,  $a = 15.32$ ,  $b = 7.59$ ,  $c = 13.16$  Å,  $\beta = 80.7^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.068$  for 1204 reflexions.

In the molecule (Fig. 1) the exocyclic  $\beta$ -naphthyloxy group is axial to the B ring, which has an envelope conformation with O(1) and O(2) almost in the plane of



M.P. GEORGIADIS, E.A. COULADOUROU, M.G. POLISSIOU, S.E. FILIPPAKIS, D. MENTZAFOS and A. TERZIS, 1982. *J. Org. Chem.*, **47**, 3054-3058.

I. Orthorhombic,  $Pbca$ ,  $a = 14.784$ ,  $b = 28.713$ ,  $c = 8.229$  Å,  $D_m = 1.35$ ,  $Z = 8$ . Mo radiation,  $R = 0.037$  for 1769 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 9.472$ ,  $b = 8.181$ ,  $c = 13.972$  Å,  $\alpha = 97.30$ ,  $\beta = 95.27$ ,  $\gamma = 67.85^\circ$ ,  $D_m = 1.33$ ,  $Z = 2$ . Mo radiation,  $R = 0.046$  for 2246 reflexions.

Both molecules (Fig. 1) have the pyran ring in sofa conformation, with ring oxygen 0.50 Å from the plane in I and 0.56 Å in II.

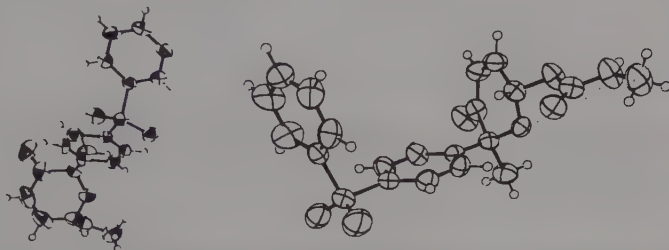


Fig. 1. Views of the pyran-3(6H)-one derivatives (I) (left) and (II) (right).

ETHYL 2,4a,4b,5,6,7,8,9,9a,10-DECAHYDRO-7,10-DIHYDROXY-2,4a-EPOXY-8-METHYLENE-7,9a-METHANOBENZ[a]AZULENE-1-CARBOXYLATE  
 $C_{19}H_{22}O_5$

M. VAN MEERSCHE, J.P. DECLERCQ, W.M. GROOTAERT and P.J. DE CLERCQ, 1982. *Bull. Soc. Chim. Belg.*, **91**, 819-824.

Monoclinic,  $P2_1/a$ ,  $a = 16.298$ ,  $b = 8.359$ ,  $c = 13.183$  Å,  $\beta = 112.32^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.051$  for 2017 reflexions.

The structure is shown in Fig. 1; the five-membered B ring is a half-chair with pseudo-diad axis through C(10). The six-membered ring of the bicyclo[3.2.1]octane group adopts a distorted chair conformation.

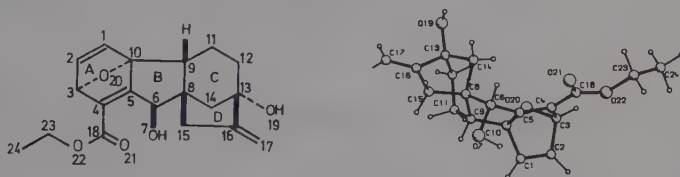


Fig. 1. The  $C_{19}H_{22}O_5$  molecule.

5a-EPI-6-OXATETRACYCLINE DIETHYL ETHER SOLVATE

$C_{20}H_{20}N_2O_8 \cdot C_4H_{10}O$

P. KOLLAT and J.J. STEZOWSKI, 1982. *Acta Cryst.*, **B38**, 2531-2533.

Triclinic,  $P\bar{1}$ ,  $a = 9.864$ ,  $b = 11.129$ ,  $c = 11.713$  Å,  $\alpha = 80.967$ ,  $\beta = 85.335$ ,  $\gamma = 70.310^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.040$  for 3498 reflexions.

The molecular structure in the crystal is that of a non-ionised free base with a short intramolecular hydrogen bond in the A-ring chromophore (Fig. 1). Bond distances are generally as expected. Crystal packing is determined by van der Waals forces.

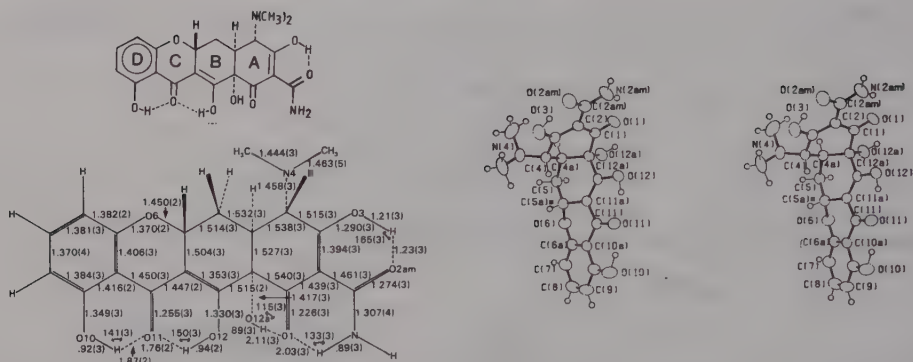


Fig. 1.  $C_{20}H_{20}N_2O_8, C_4H_{10}O$ : stereoscopic view and molecular structure, with bond distances and also bond angles for H atoms involved in intramolecular hydrogen bonds.

3-(1,4-EPOXY-2-HYDROXY-2,6,6-TRIMETHYLCYCLOHEXYL)-1-METHYL-2-PROPYL *p*-NITROBENZOATE  
 $C_{20}H_{25}NO_6$

Y. KITANO, T. KATO, H. KONDO and T. ASHIDA, 1982. Bull. Chem. Soc. Jpn., 55, 3429-3433.

Triclinic,  $P\bar{1}$ ,  $a = 11.618$ ,  $b = 12.373$ ,  $c = 7.934$  Å,  $\alpha = 108.43$ ,  $\beta = 108.89$ ,  $\gamma = 95.95^\circ$ ,  $D_m = 1.24$ ,  $Z = 2$ . Cu radiation,  $R = 0.073$  for 2550 reflexions.

The analysis established the relative configuration at C9 to be as shown in Fig. 1.

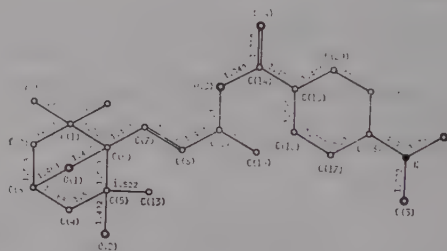
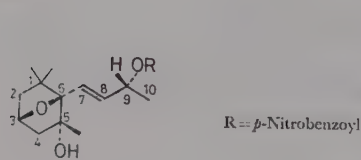


Fig. 1. The  $C_{20}H_{25}NO_6$  molecule and bond lengths.

3,14-DIMETHYL-5,11,16,22-TETRAOXO-1,4,12,15-TETRAOXACYCLODOCOSANE  
 $C_{20}H_{32}O_8$

A. SHANZER, J. LIBMAN, H. GOTTLIEB and F. FROLOW, 1982. J. Am. Chem. Soc., 104, 4220-4225.

Monoclinic,  $P2_1/c$ ,  $a = 13.437$ ,  $b = 7.680$ ,  $c = 10.997$  Å,  $\beta = 107.08^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.078$  for 1151 reflexions.

The two methyl substituents of the centrosymmetric heterocyclic molecule (Fig. 1) are located at diagonal positions, assuming an 'anti' configuration. No unusual geometrical parameters were noted.

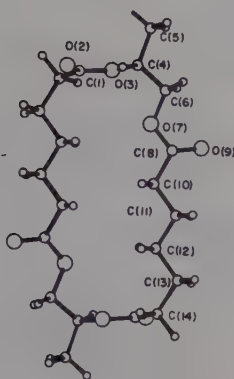


Fig. 1. The  $C_{20}H_{32}O_8$  molecule.

5'-BROMO-6',7'-DIHYDRO-3'-METHOXYCARBONYL-4-PHTHALIMIDOMETHYL-SPIRO[1,3-DIOXOLANE-2,4'-(5'H)-BENZO[b]FURAN]

$C_{21}H_{18}BrNO_7$  (I and II)

M. SAKAI, H. NAKAI and M. SHIRO, 1982. Cryst. Struct. Comm., **11**, 403-407.

I. Monoclinic,  $P2_1/c$ ,  $a = 14.572$ ,  $b = 9.363$ ,  $c = 21.439$  Å,  $\beta = 136.47^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.051$  for 3082 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 8.228$ ,  $b = 16.708$ ,  $c = 7.704$  Å,  $\alpha = 98.72$ ,  $\beta = 91.48$ ,  $\gamma = 76.17^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.048$  for 3185 reflexions.

The analysis was undertaken to determine the relative configuration of the asymmetric centres; these are: 2R,4R,5'R for (I) and 2R,4S,5'R for (II) (Fig. 1).

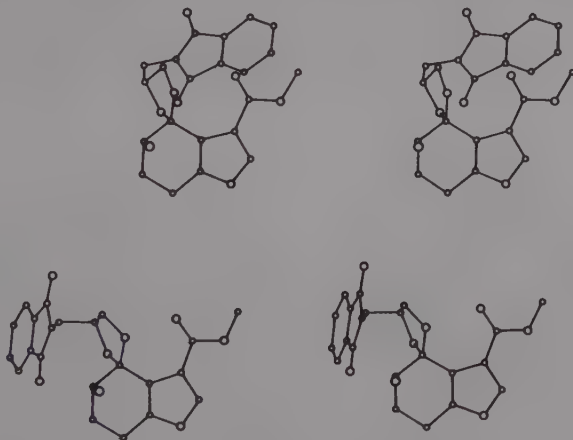


Fig. 1. Stereoviews of the two isomers of  $C_{21}H_{18}BrNO_7$ ; (I) (top) and (II) (bottom).



## DIHYDROHERQUEINONE MONOMETHYL ETHER

 $C_{21}H_{24}O_7$ 

T. YOSHIOKA, T. HIRATA, T. AOKI and T. SUGA, 1982. Bull. Chem. Soc. Jpn., 55, 3847-3851.

Monoclinic,  $P2_1$ ,  $a = 11.213$ ,  $b = 6.229$ ,  $c = 14.252$  Å,  $\beta = 110.21^\circ$ ,  $D_m = 1.40$ ,  $Z = 2$ . Mo radiation,  $R = 0.058$  for 1811 reflexions. Cu radiation,  $R = 0.0662$  for 2784 reflexions.

The structure and absolute stereochemistry are shown in Fig. 1. There are three intramolecular O-H...O hydrogen bonds.

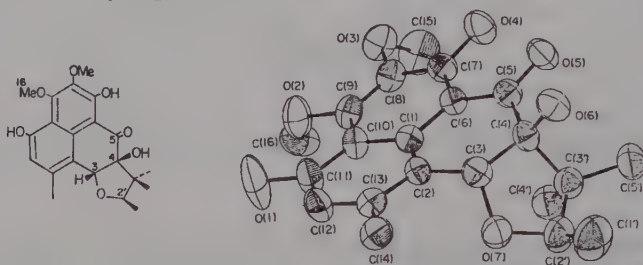


Fig. 1. The  $C_{21}H_{24}O_7$  molecule.

## 3-BENZOYL-5,8-DIHYDROXYFLAVONE

 $C_{22}H_{14}O_5$ 

H.W. SCHMALLE, O.H. JARCHOW, B.M. HAUSEN and K.-H. SCHULZ, 1982. Acta Cryst., B38, 3163-3165.

Monoclinic,  $P2_1/c$ ,  $a = 10.149$ ,  $b = 18.124$ ,  $c = 9.393$  Å,  $\beta = 100.54^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.082$  for 1288 reflexions.

The benzo-4-pyrone system (Fig. 1) is nearly planar. The conformation of the molecule is characterized by three planes: the A, B rings, the phenyl ring C and the benzoyl ring D. The angle between rings A and C is  $32(2)^\circ$ , A and D  $82(2)^\circ$ , and C and D  $113(1)^\circ$ . The hydroxyl H at O(5) is hydrogen-bonded to the C(4) carbonyl group. Intermolecular hydrogen bonds of the type O-H...O form endless chains with alternate molecules along b.

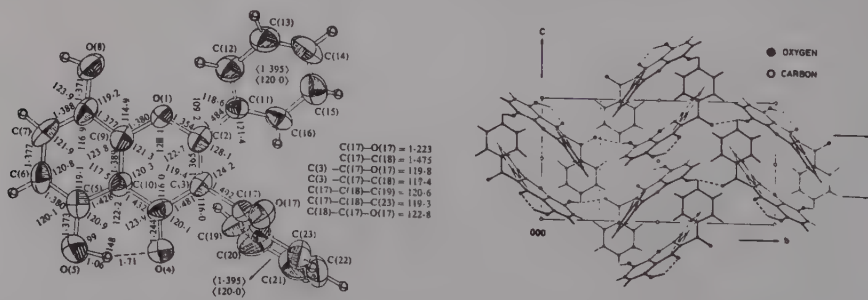


Fig. 1. The  $C_{22}H_{14}O_5$  molecule with dimensions, and a packing diagram.

3,15-DIMETHYL-18-(1-PROPYNYL)-16-OXAPENTACYCLO[10.4.2.0<sup>2,11</sup>.0<sup>3,8</sup>.0<sup>15,18</sup>]OCTADEC-4,7-DIEN-6-ONE $C_{22}H_{26}O_2$

E.F. PAULUS and G.M. SHELDRICK, 1982. *Acta Cryst.*, B38, 284-286.

Monoclinic,  $P2_1$ ,  $a = 20.383$ ,  $b = 7.316$ ,  $c = 12.094$  Å,  $\beta = 90.74^\circ$ ,  $D_m = 1.18$ ,  $Z = 4$ . Mo radiation,  $R = 0.056$  for 2902 reflexions.

In the crystal structure the two independent molecules in the asymmetric unit are virtually identical and have a steroid-like arrangement of three six- and one five-membered rings, with an additional transannular ether linkage (Fig. 1). Bond lengths and angles are normal.

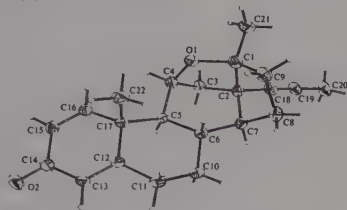


Fig. 1. A perspective view of one of the two independent  $C_{22}H_{26}O_2$  molecules.

BIS(3-ETHOXYSELENOFORMYLOXY-2,2,5,5-TETRAMETHYL-2H,5H-DIHYDROFURANYL)DISELENIDE  
 $C_{22}H_{34}O_6Se_4$

J.S. LEE, S.C. NYBURG and K. PRAEFCKE, 1982. *Chem. Zeit.*, 106, 375-377.

Monoclinic,  $P2_1/c$ ,  $a = 15.216$ ,  $b = 11.596$ ,  $c = 17.537$  Å,  $\beta = 107.84^\circ$ ,  $D_m = 1.60$ ,  $Z = 4$ . Cu radiation,  $R = 0.067$  for 3126 reflexions.

The molecule has a twofold axis of symmetry, along the Se(1)-Se(2) bond (Fig. 1). The Se(1)-Se(2) distance is 2.342 Å and the Se(1)-CA1 and Se(2)-CB1 bond lengths are 1.898 and 1.884 Å respectively. The C=Se double bond distances CA52-Se(3) and CB52-Se(4) are respectively 1.79 and 1.76 Å and the conformation of the molecule is such that the intramolecular Se(1)...Se(3) and Se(2)...Se(4) distances are 3.74 and 3.79 Å respectively compared to the intermolecular interactions of 3.98 Å (Se(1)...Se(4')) and 3.77 Å (Se(2)...Se(4')).

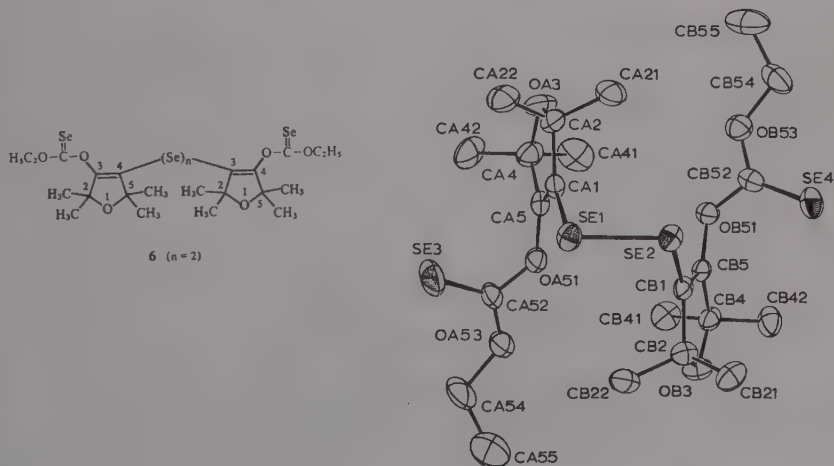


Fig. 1. Molecular structure of  $C_{22}H_{34}O_6Se_4$ .

3,3-DIMETHYL-2-ETHOXYCARBOXYLATO-7,7-ETHYLENEDIOXY-8-HYDROXYMETHYL-6-PIVALATO-METHYL-BICYCLO[3.3.0]OCTANE

$C_{22}H_{36}O_8$

P.F. SCHUDA, H.L. AMMON, M.R. HEIMANN and S. BHATTACHARJEE, 1982. J. Org. Chem., 47, 3434-3440.

Triclinic,  $P\bar{1}$ ,  $a = 6.733$ ,  $b = 13.33$ ,  $c = 14.65$  Å,  $\alpha = 71.07$ ,  $\beta = 82.42$ ,  $\gamma = 78.10^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.114$  for 2581 reflexions.

The analysis established the structure and stereochemistry as shown in Fig. 1. The C-7 carbon is above the plane formed by the other four carbon atoms of the B ring, and the C-11 carbon is below the plane formed by the four carbons of the C ring. A least-squares planes analysis shows that C-7 is 0.66 Å above the plane formed by C-2, C-3, C-8, and C-9, and C-11 is 0.70 Å below the plane formed by C-1, C-2, C-9, and C-10.

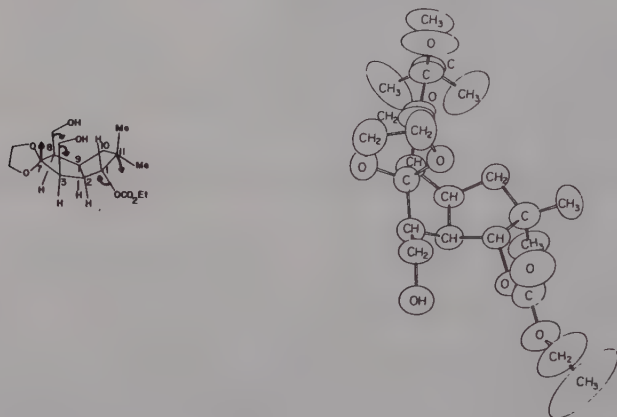


Fig. 1. The  $C_{22}H_{36}O_8$  molecule.

S-t-BUTYLTHIOURONIUM PERCHLORATE 1,4,7,10,13,16-HEXAOXACYCLOOCTADECANE (2:1)  
 $C_{22}H_{50}Cl_2N_4O_{14}S_2$   $C_{12}H_{24}O_6 \cdot 2C_5H_{13}N_2S^+ \cdot 2ClO_4^-$

J.W.H.M. UITERWIJK, S. HARKEMA, G.J. VAN HUMMEL, J. GEEVERS and D.N. REINHOUDT, 1982. Acta Cryst., B38, 1862-1864.

Triclinic,  $P\bar{1}$ ,  $a = 10.905$ ,  $b = 10.722$ ,  $c = 9.329$  Å,  $\alpha = 111.41$ ,  $\beta = 98.47$ ,  $\gamma = 110.40^\circ$ ,  $Z = 1$ . Mo radiation,  $R = 0.048$  for 2524 reflexions.

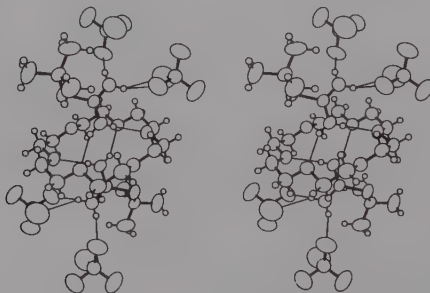


Fig. 1. Stereoview of the  $C_{22}H_{50}Cl_2N_4O_{14}S_2$  complex.

The macrocyclic ring has an almost ideal "crown" conformation with approximate  $D_{3d}$  symmetry (Fig. 1). One of the  $NH_2$  groups is hydrogen bonded to two O atoms of the ring (next nearest neighbours) with  $N...O$  2.898 and 2.972(5) Å while the other forms two hydrogen bonds, one bifurcated, to two perchlorate ions ( $N...O$  3.104, 3.225, 3.031(5) Å). Mean bond lengths and angles are, C-C 1.481(3), C-O 1.422(2) Å, O-C-C 109.6(2), C-O-C 112.3(2)°.

2,3,3a,7,8,8a,10,11,12,13b-DECAHYDRO-5,7,11,11-TETRAMETHYL-7,13c-EPOXY-13cH-BENZO[c]-FURO[3',2':6,7]CYCLOHEPTA[1,2,3-ij][2]BENZOPYRAN-13(1H)-ONE  
 $C_{23}H_{28}O_4$

A.L. SPEK and A.J.M. DUISENBERG, 1982. Cryst. Struct. Comm., 11, 417-422.

Triclinic,  $P\bar{1}$ ,  $a = 9.220$ ,  $b = 10.138$ ,  $c = 12.790$  Å,  $\alpha = 101.22$ ,  $\beta = 113.16$ ,  $\gamma = 109.19^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.042$  for 2387 reflexions.

The analysis established the structure of this reaction product to be as shown in Fig. 1.

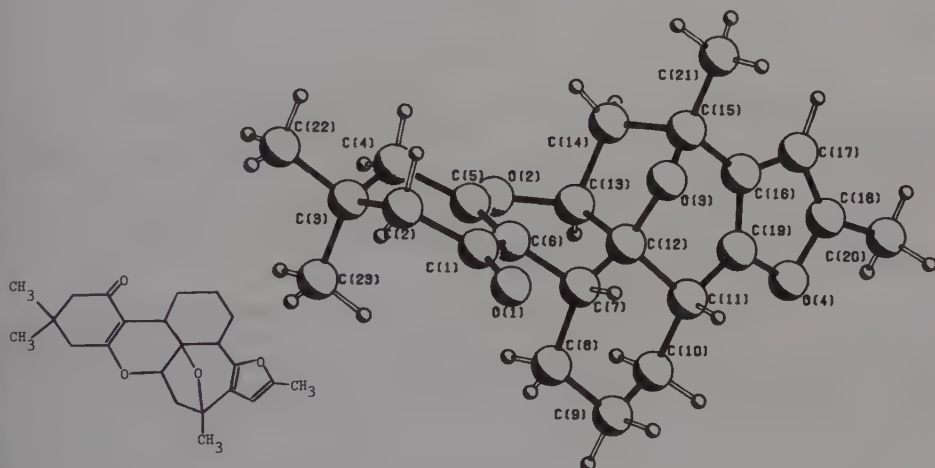


Fig. 1. The  $C_{23}H_{28}O_4$  molecule.

2-PROPIONYLETHYLAMINO-3-METHYL-3-(2'-HYDROXY-5'-ACETOXY)PHENYL-5-ACETOXY-2,3-DIHYDRO-BENZOFURAN  
 $C_{24}H_{27}NO_7$

Z. URBANCZYK-LIPKOWSKA, J.W. KRAJEWSKI, P. GLUZINSKI, L. KOZERSKI and M. CZUGLER, 1982. Cryst. Struct. Comm., 11, 1531-1536.

Triclinic,  $P\bar{1}$ ,  $a = 10.596$ ,  $b = 14.186$ ,  $c = 7.765$  Å,  $\alpha = 105.48$ ,  $\beta = 95.55$ ,  $\gamma = 83.93^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.058$  for 3026 reflexions.

The conformation of the furan ring (Fig. 1) is intermediate between envelope and twist forms. The OAc side chain at the benzofuran ring is disordered over two sites. Molecules are linked into centrosymmetric hydrogen bonded dimers, with 18-membered rings, by O(9)-H...O(30) hydrogen bonds (O...O 2.681(3) Å).

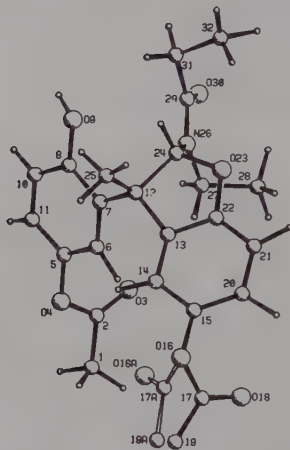


Fig. 1. A view of  $C_{24}H_{27}NO_7$ .

4-NITRO-1,2-BENZENEDIAMINE 1,4,7,10,13,16-HEXAOXACYCLOOCTADECANE(18-CROWN-6) 2:1  
COMPLEX

$C_{24}H_{38}N_6O_{10}$

$2C_6H_7N_3O_2 \cdot C_{12}H_{24}O_6$

G. WEBER, 1982. Acta Cryst., B38, 629-632.

Monoclinic,  $P2_1/c$ ,  $a = 9.740$ ,  $b = 10.178$ ,  $c = 14.615$  Å,  $\beta = 95.12^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.047$  for 2101 reflexions.

The two centrosymmetrically related 4-nitro-1,2-benzenediamine molecules are inclined at  $120^\circ$  above and below the plane respectively of the six ether oxygen atoms to which they are linked by non-bifurcated hydrogen bonds (Fig. 1). The macrocycle has a conformation with a sequence of unique torsion angles  $ag^+a$ ,  $ag^+a$ ,  $g^+g^+a$  and average dimensions, C-C 1.493, C-O 1.419 Å, C-O-C 112.6, O-C-C 109.3° (neglecting O(1)-C(2)-C(3) 114.0°).

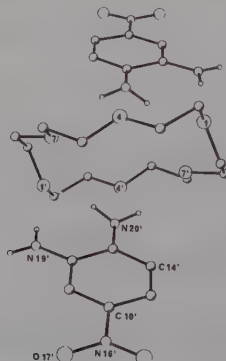
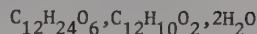
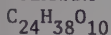


Fig. 1. The  $C_{24}H_{38}N_6O_{10}$  adduct with dotted lines indicating hydrogen bonds.

1,4,7,10,13,16-HEXAOXACYCLOCTADECANE(18-CROWN-6) 4,4'-BIPHENYLDIOL DIHYDRATE  
TERNARY COMPLEX

D.A. GROSSIE, W.H. WATSON, F. VOGTLE and W.M. MULLER, 1982. *Acta Cryst.*, **B38**, 3157-3159.

Monoclinic,  $P2_1/c$ ,  $a = 9.264$ ,  $b = 11.007$ ,  $c = 14.165$  Å,  $\beta = 109.10^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.073$  for 1138 reflexions.

The 18-crown-6 ether (Fig. 1) sits on a centre of symmetry. The six O atoms are alternately 0.23(2) Å above and below the mean plane and form an approximate hexagon with averaged sides of 2.83(1) Å. The two water molecules are each hydrogen bonded to two O atoms of the crown ether. The planar 4,4'-biphenyldiol molecules sit on centres of symmetry and are hydrogen-bonded to the water molecules. The 4,4'-biphenyldiol molecules bridge two crown ethers via hydrogen bonds to water molecules.

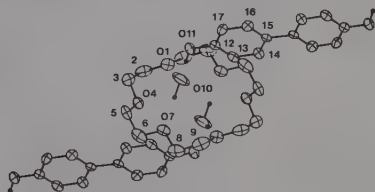


Fig. 1. A view of the ternary complex.

## (+)-(6aSR,11aSR,11bRS)-1,6a,11a,11b-TETRAHYDRO-5,7,8-TRIMETHOXY-3,6a,10,11b-TETRA-METHYLDIINDENO[7,1-bc:2,1-e]PYRAN-2,11-DIONE



J.L. FLIPPEN-ANDERSON and F.D. MILLS, 1982. *Acta Cryst.*, **B38**, 495-498.

Triclinic,  $P\bar{1}$ ,  $a = 10.093$ ,  $b = 13.811$ ,  $c = 15.916$  Å,  $\alpha = 77.6$ ,  $\beta = 82.7$ ,  $\gamma = 87.5^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.089$  for 5601 reflexions.

Both molecules in the asymmetric unit have the same conformation. The C(9), C(11), C(19), O(1) bridging plane forms dihedral angles of 59.8(5) and 48.2(5)° with the AB and A'B' ring systems (Fig. 1) which, excluding C(29), C(30) and C(31), are each planar. (For molecule II the angles are 56.5(5) and 43.4(5)°). The methoxy groups are coplanar with their attached rings except for C(29) which is denied coplanarity by a hydrogen atom on C(31). The H at C(11) is cis with respect to C(31) and gauche with respect to C(30).

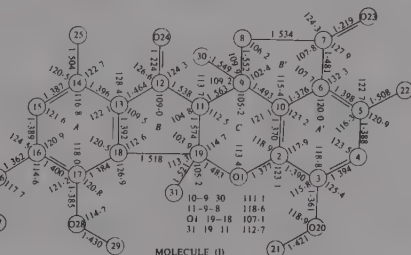


Fig. 1. Bond lengths and angles in the  $C_{25}H_{26}O_6$  molecule.



3,6,9,12,15,18,21,26-OCTAOXABICYCLO[21.2.1]HEXACOSA-1(25),23-DIENE-2,22-DIONE  
BENZYLAMMONIUM PERCHLORATE

$C_{25}H_{36}ClNO_{14}$

$C_{18}H_{26}O_{10} \cdot C_7H_{10}N^+ \cdot ClO_4^-$

N.K. DALLEY, J.S. BRADSHAW, S.B. LARSON and S.H. SIMONSEN, 1982. *Acta Cryst.*, **B38**, 1859-1862.

Monoclinic,  $P2_1/c$ ,  $a = 11.710$ ,  $b = 15.087$ ,  $c = 17.574$  Å,  $\beta = 105.68^\circ$ ,  $D_m = 1.334$ ,  $Z = 4$ . Mo radiation,  $R = 0.037$  for 3645 reflexions (at 173 K).

The macrocyclic ligand is hydrogen bonded to the organic cation and is wrapped about it such that the benzene ring is sandwiched between the furan ring (dihedral angle  $37^\circ$ ) and that part of the polyether opposite the furan ring (Fig. 1). The cation is hydrogen bonded to the perchlorate anion via two hydrogen bonds. Molecular geometry is normal.

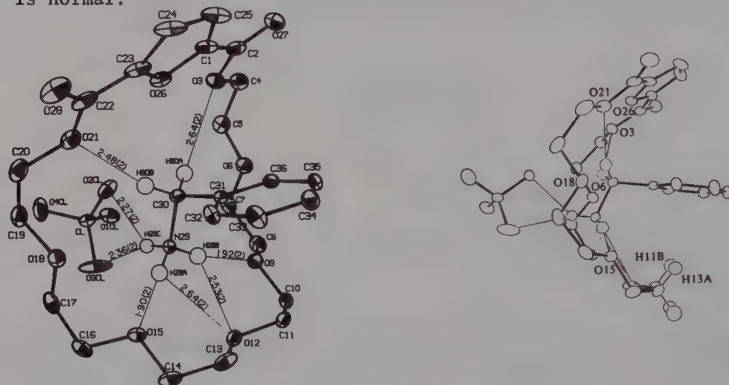
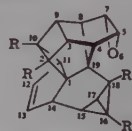


Fig. 1. Two views of the  $C_{25}H_{36}ClNO_{14}$  complex.

(DL)-(1R,5S,11S,14R,16S)-6-OXADECACYCLO[9.8.0.0<sup>1,14</sup>.0<sup>2,10</sup>.0<sup>3,9</sup>.0<sup>4,19</sup>.0<sup>5,7</sup>.0<sup>8,19</sup>.0<sup>15,17</sup>.0<sup>16,18</sup>]NONADEC-12-EN-2,10,16,18-TETRACARBOXYLIC ACID TETRAMETHYL ESTER

$C_{26}H_{24}O_9$



K.-H. LEHR, R. HILDEBRAND, H. FRITZ, L. KNOTHE, C. KRUGER and H. PRINZBACH, 1982. *Chem. Ber.*, **115**, 1875-1901.

Monoclinic,  $P2_1/a$ .  $R = 0.046$  for 4750 reflexions.

The structure was established as that shown above. Bond lengths and angles have expected values. All crystal data are deposited.

2-PROPIONYLETHYLAMINO-3-METHYL-3-(2',5'-DIACETOXY)PHENYL-5-ACETOXY-2,3-DIHYDROBENZO-FURAN

$C_{26}H_{29}NO_8$

Z. URBANCZYK-LIPDOWSKA, J.W. KRAJEWSKI, P. GLUZINSKI, G.D. ANDREETTI and G. BOCELLI, 1982. *Cryst. Struct. Comm.*, **11**, 1537-1542.

Monoclinic,  $P2_1/n$ ,  $a = 16.310$ ,  $b = 19.235$ ,  $c = 8.127$  Å,  $\beta = 98.59^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.068$  for 3640 reflexions.

In the molecule (Fig. 1) the values of the torsional angles around the bond involving the two asymmetric carbon atoms in the furan ring C6-C16-C24-N29 and C15-C16-C24-N29 ( $-22.1(5)^\circ$  and  $-145.6(4)^\circ$ , respectively) indicate that the bulky substituents are in Z configuration. The furan ring conformation is between envelope and twist conformations.

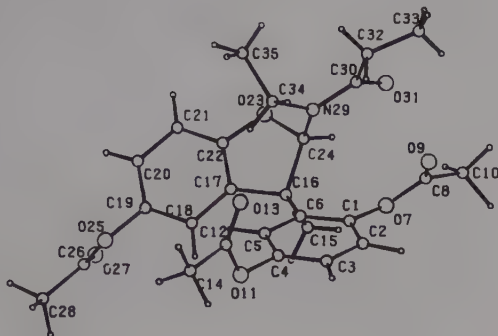


Fig. 1. A view of  $C_{26}H_{29}NO_8$ .

1,4,7,10,13,16-HEXAOXACYCLOOCTADECANE PHENYL CARBAMATE  
 $C_{26}H_{38}N_2O_{10}$

$C_{12}H_{24}O_6 \cdot 2C_7H_7NO_2$

J. GALLOY, W.H. WATSON, F. VÖGTLE and W.M. MUELLER, 1982. Acta Cryst., **B38**, 1245-1248.

Monoclinic,  $P2_1/c$ ,  $a = 10.053$ ,  $b = 11.893$ ,  $c = 12.359$  Å,  $\beta = 105.51^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.040$  for 1518 reflexions.

In the crystal structure (Fig. 1) the 18-crown-6 ether sits on a centre of symmetry and exhibits approximate  $D_{3d}$  symmetry. The six O atoms are alternately 0.24 Å above and below the mean plane and form a nearly regular hexagon with averaged sides of 2.86 Å. The two guest molecules are related by a centre of symmetry and each forms hydrogen bonds, with H...O distances of 2.12 and 2.39 Å.

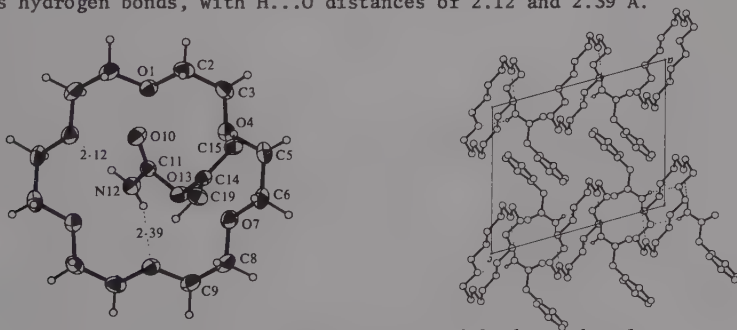


Fig. 1.  $C_{26}H_{38}N_2O_{10}$ : a view of the complex, with three phenyl atoms omitted for clarity (left) and a packing diagram showing hydrogen bonding (right).

3,7-DI-*t*-BUTYL-5-PHENYL-2-(*p*-TOLYL)OXEPIN  
 $C_{27}H_{32}O$

A. RIEKER, S. BERGER, D. MOOTZ, M. STEFFEN and H. WUNDERLICH, 1982. Chem. Ber., **115**, 385-389.

Triclinic,  $P\bar{1}$ ,  $a = 8.902$ ,  $b = 11.748$ ,  $c = 11.598$  Å,  $\alpha = 104.87$ ,  $\beta = 98.41$ ,  $\gamma = 73.82^\circ$ ,  $D_m = 1.10$ ,  $Z = 2$ . Cu radiation,  $R = 0.103$  for 2783 reflexions.

The seven-membered heterocycle (Fig. 1) adopts a boat conformation with the O at the bow and C(4) and C(5) the stern. The dihedral angles between the C(2),C(3),C(6),C(7) mean plane and planes C(2),O,C(7) and C(3),C(4),C(5),C(6) are  $117.6$  and  $150.7^\circ$  respectively. There is some delocalization in this ring; the lengths of the formal single and double bonds lie in the range  $1.323(3)$ – $1.441(3)$  Å.

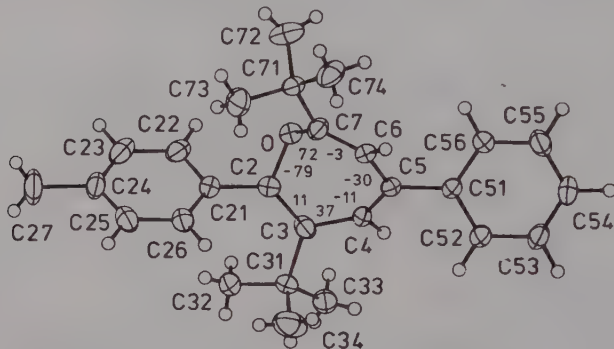


Fig. 1. The structure of  $C_{27}H_{32}O$  showing the torsion angles ( $^\circ$ ) in the seven-membered ring.

(21R,26S,29S,34R)-21,22,23,24,25,26,29,30,31,32,33,34-DODECAHYDRO-1,4,7,14,17,20,28,35-OCTAOXA[2<sup>3,29</sup>.2<sup>18,34</sup>][7.7]ORTHOCYCLOPHANE (ISOMER A)

$C_{28}H_{42}O_8$  (I)

(21R,26S,29R,34S)-21,22,23,24,25,26,29,30,31,32,33,34-DODECAHYDRO-1,4,7,14,17,20,28,35-OCTAOXA[2<sup>3,29</sup><sub>syn</sub>.2<sup>18,34</sup><sub>syn</sub>][7.7]ORTHOCYCLOPHANE (ISOMER B)

$C_{28}H_{42}O_8$  (II)

(21R,26S,29R,34S)-21,22,23,24,25,26,29,30,31,32,33,34-DODECAHYDRO-1,4,7,14,17,20,28,35-OCTAOXA[2<sup>3,29</sup><sub>syn</sub>.2<sup>18,34</sup><sub>syn</sub>][7.7]ORTHOCYCLOPHANE (ISOMER B) POTASSIUM PERCHLORATE (1:1 COMPLEX)

$C_{28}H_{42}ClKO_{11}$  (III)

$C_{28}H_{42}O_8.KClO_3$

J.A. BANDY and M.R. TRUTER, 1982. *Acta Cryst.*, **B38**, 2639-2648.

I. Monoclinic,  $P2_1/a$ ,  $a = 15.613$ ,  $b = 21.492$ ,  $c = 8.021$  Å,  $\beta = 96.99^\circ$ ,  $D_m = 1.26$ ,  $Z = 4$ . Mo radiation,  $R = 0.038$  for 2163 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 8.842$ ,  $b = 16.702$ ,  $c = 10.050$  Å,  $\alpha = 114.10$ ,  $\beta = 98.97$ ,  $\gamma = 91.22^\circ$ ,  $D_m = 1.28$ ,  $Z = 2$ . Mo radiation,  $R = 0.035$  for 2666 reflexions.

III. Monoclinic,  $P2_1/c$ ,  $a = 10.990$ ,  $b = 14.317$ ,  $c = 19.583$  Å,  $\beta = 90.64^\circ$ ,  $D_m = 1.39$ ,  $Z = 4$ . Mo radiation,  $R = 0.074$  for 2795 reflexions.

In isomer A (I) (Fig. 1) the cis H atoms on the two cyclohexane/14-crown-4 ring junctions are on opposite sides (relative configuration 21R, 26S, 29S and 34R). In isomer B (II) (Fig. 1) the cis H atoms on the two junctions are on the same side (relative configuration 21R, 26S, 29R and 34S) and opposite to that of the  $OCH_2CH_2OC_6H_4OCH_2CH_2O$  bridge. In the 1:1 complex of isomer B with potassium perchlorate (III) (Fig. 1) the  $K^+$  ion is coordinated by seven atoms of the ligand with K-O in the range  $2.670$ – $2.853(4)$  Å, and one O atom of the perchlorate ion at  $2.922(10)$  Å; the eighth O atom of the ligand is at  $3.145(4)$  Å from the  $K^+$  ion. The complexed isomer B (III) differs in conformation from that of the uncomplexed form.

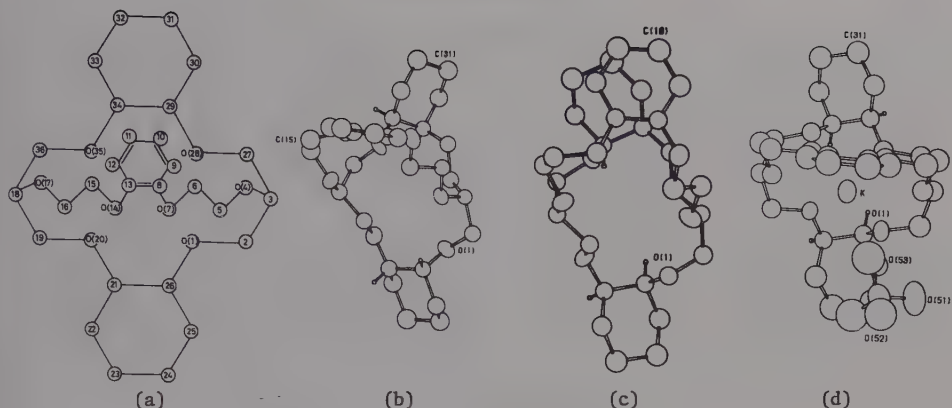


Fig. 1.  $C_{28}H_{42}O_8$ : (a) structure of the cyclophane, (b) isomer A (I), (c) isomer B (II), (d) complexed isomer B (III),

(21S,26R,29S,34R)-21,22,23,24,25,26,29,30,31,32,33,34-DODECAHYDRO-1,4,7,14,17,20,28,35-OCTAOXA[2<sup>3,29</sup>anti.2<sup>18,34</sup>anti][7.7]ORTHOCYCLOPHANE (ISOMER C)

$C_{28}H_{42}O_8$  (I)

(21S,26R,29S,34R)-21,22,23,24,25,26,29,30,31,32,33,34-DODECAHYDRO-1,4,7,14,17,20,28,35-OCTAOXA[2<sup>3,29</sup>anti.2<sup>18,34</sup>anti][7.7]ORTHOCYCLOPHANE (ISOMER C) METHANOL (1:1 COMPLEX)

$C_{29}H_{46}O_9$  (II)

$C_{28}H_{42}O_8 \cdot CH_3OH$

J.A. BANDY, D.L. HUGHES and M.R. TRUTER, 1982. *Acta Cryst.*, B38, 2648-2657.

I. Monoclinic,  $P2_1/a$ ,  $a = 14.832$ ,  $b = 12.806$ ,  $c = 14.555$  Å,  $\beta = 103.88^\circ$ ,  $D_m = 1.25$ ,  $Z = 4$ . Mo radiation,  $R = 0.036$  for 2546 reflexions (at 295 K).

II. Monoclinic,  $P2_1/n$ ,  $a = 14.520$ ,  $b = 15.391$ ,  $c = 12.754$  Å,  $\beta = 95.02^\circ$ ,  $D_m = 1.26$ ,  $Z = 4$ . Mo radiation,  $R = 0.040$  for 2071 reflexions (at 295 K);  $a = 14.353$ ,  $b = 15.279$ ,  $c = 12.659$  Å,  $\beta = 95.00^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.033$  for 2060 reflexions (at 123 K).

The macrobicyclic molecule consists of a dicyclohexano-14-crown-4 ring bridged by an  $OC_2H_2OC_6H_4OC_2H_2O$  group (Fig. 1). In isomer C the four cis H atoms at the cyclohexano ring junctions are on the same side of the 14-crown-4 entity as the bridge. The conformation of the molecule is essentially unchanged in the methanol complex where the methanol hydroxy group forms a weak bifurcated hydrogen bond to the O atoms on the aromatic ring.

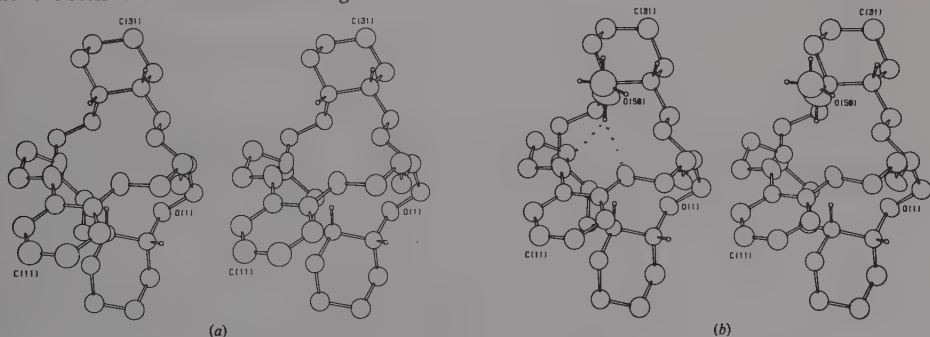


Fig. 1.  $C_{28}H_{42}O_8$ : stereoscopic views of (a) isomer C (I) and (b) the complex of I with methanol (II) (right).

BIS(17-HYDROXY-15(O<sub>5</sub>-(1,2-BENZENO.2<sub>4</sub>-CORONAND-5)))POTASSIUM THIOCYANATE  
 $C_{29}H_{40}KNO_{12}S$   $[C_{28}H_{40}O_{12}]K^+, SCN^-$

E. WEBER and M. CZUGLER, 1982. *Inorg. Chim. Acta*, **61**, 33-38.

Triclinic,  $P\bar{1}$ ,  $a = 9.615$ ,  $b = 11.572$ ,  $c = 14.893$  Å,  $\alpha = 91.63$ ,  $\beta = 95.06$ ,  $\gamma = 96.13^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.100$  for 2311 reflexions.

The structure was refined initially in space group  $P\bar{1}$  and then in the non-centrosymmetric one, to account partially for the disorder present. There are two independent molecules in the unit cell (Fig. 1). The  $SCN^-$  anions are well separated from the  $K^+$  cations which are sandwiched between a pair of crown rings. Each cation is tenfold coordinated with mean  $K^+ \dots O$  distances of 2.89(6) Å (K(1)) and 2.90(9) Å (K(2)).

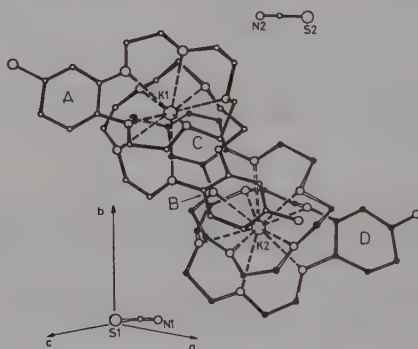


Fig. 1. View of the crystal structure of  $C_{29}H_{40}KNO_{12}S$ .

(R)-3,7,10,14,17,21,24,28,31,35-PENTAKIS(EPOXY)-1,8,15,22,29-PENTAOXACYCLOPENTATRIACONTANE-2,9,16,23,30-PENTAONE ACETONITRILE SOLVATE  
 $C_{30}H_{40}O_{15} \cdot C_2H_3N$  (I)

(R,S)-3,7,10,14,17,21,24,28,31,35-PENTAKIS(EPOXY)-1,8,15,22,29-PENTAOXACYCLOPENTATRIACONTANE-2,9,16,23,30-PENTAONE ACETONE SOLVATE  
 $C_{30}H_{40}O_{15} \cdot C_3H_6O$  (II)

I. SAKURAGI, I. TANAKA, T. ASHIDA, I. TAJIMA, M. OKADA and H. SUMITOMO, 1982. *J. Am. Chem. Soc.*, **104**, 6035-6039.

I. Orthorhombic,  $P2_12_12_1$ ,  $a = 10.050$ ,  $b = 14.764$ ,  $c = 24.268$  Å,  $D_m = 1.28$ ,  $Z = 4$ . Mo radiation,  $R = 0.083$  for 2355 reflexions.

II. Orthorhombic,  $P2_12_12_1$ ,  $a = 9.863$ ,  $b = 14.727$ ,  $c = 24.335$  Å,  $D_m = 1.29$ ,  $Z = 4$ . Cu radiation,  $R = 0.077$  for 2662 reflexions.

The two structures (Fig. 1) are isomorphous with one another and the pentamer, which is a nearly regular pentagonal shaped molecule, includes an acetonitrile or acetone molecule in its cavity. The solvent molecules are so aligned that their dipole moments are opposite to that of the pentamer molecule.



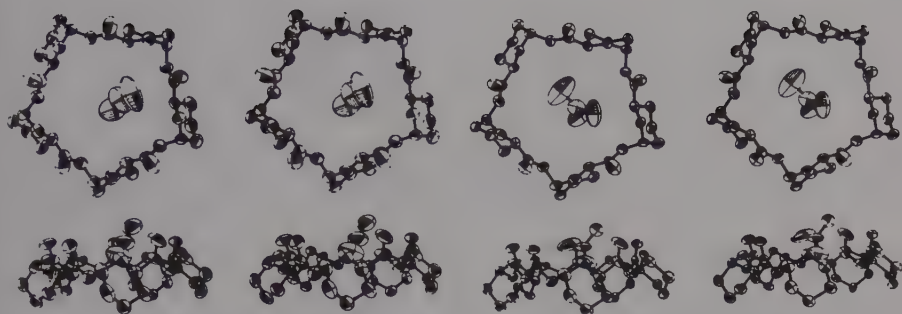


Fig. 1. Stereoviews of  $C_{30}H_{40}O_{15}, C_2H_3N$  (left) and  $C_{30}H_{40}O_{15}, C_3H_6O$  (right).

3',8-BIS[(p-CHLOROPHENYL)THIOMETHOXY]-4',5,6',7-TETRAMETHYLCHROMAN-2-SPIRO-1'-CYCLOHEXA-3',5'-DIEN-2'-ONE

$C_{32}H_{30}Cl_2O_4S_2$

P. CACIOLI, M.F. MACKAY and J.A. REISS, 1982. *Acta Cryst.*, **B38**, 1001-1004.

Triclinic,  $P\bar{1}$ ,  $a = 7.636$ ,  $b = 14.167$ ,  $c = 15.186$  Å,  $\alpha = 108.75$ ,  $\beta = 100.54$ ,  $\gamma = 77.41^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.065$  for 2534 reflexions.

In the molecule (Fig. 1) the conformation and dimensions of the pyranspiro-cyclohexadienone moiety are similar to those observed in the 3',8-bis(chloromethoxy) analogue (1). The pyran ring system is in a half-chair form and the cyclohexadienone ring is envelope. In the 1,3-diene moiety, the two long and two short C-C bonds have mean values of 1.461 and 1.332 Å respectively.

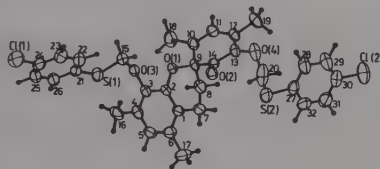


Fig. 1.  $C_{32}H_{30}Cl_2O_4S_2$ : a perspective view of the molecule.

1. *Structure Reports*, **48B**, 383.

TETRAPHENYLDIPYRANYLIDENE

$C_{34}H_{24}O_2$

D. CHASSEAU, J. GAULTIER, C. HAUW, R. FUGNITTO, V. GIANIS and H. STRZELECKA, 1982. *Acta Cryst.*, **B38**, 1629-1631.

Monoclinic,  $P2_1/c$ ,  $a = 6.046$ ,  $b = 17.070$ ,  $c = 14.139$  Å,  $\beta = 123.44^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.043$  for 631 reflexions.

The centrosymmetric molecules pack in columns along  $a$  with adjacent stacks of parallel molecules in the  $c$  direction forming (010) sheets. While the molecules are almost planar, the heterocyclic ring has a slight boat deformation with O(18) and C(3) 0.045 and 0.021 Å respectively from the C(1), C(2), C(4), C(5) plane. Average bond lengths (Fig. 1) are C(1)-O 1.389(7), C(1)-C(2) 1.337(8), C(2)-C(3) 1.443(8) Å.



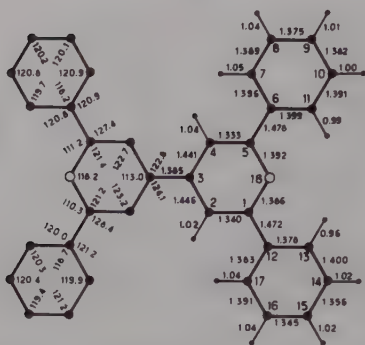


Fig. 1. The tetraphenyldipyranylidene molecule with bond lengths and angles.

12-BENZOATO-2-((1,5-DIMETHYLHEXYL-6-METHYLBICYCLO[4.3.0]NONANE)-2-SPIRO-2'-(1-OXACYCLOPROP-3-YL))-1,9-EPOXY-5-PHENYL-3,5,7-TRIAZATRICYCLO[7.4.0.0<sup>3,7</sup>]TRIDECA-4,6-DIONE

$C_{34}H_{48}N_3O_5$

C. KRATKY, W. REISCHL, E. ALTMANN and E. ZBIRAL, 1982. *Monatsh. Chem.*, **113**, 439-448.

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.480$ ,  $b = 14.081$ ,  $c = 36.38$  Å,  $Z = 4$ . Mo radiation,  $R = 0.09$  for 1399 reflexions.

The crystal structure analysis proved that the compound was indeed the (5R,10S)-(7R,8R)-dioxirane of the  $\beta$ -isomer (6S)-4-phenyl-1,2,4-triazolin-3,5-dione adduct of vitamin D (Fig. 1).

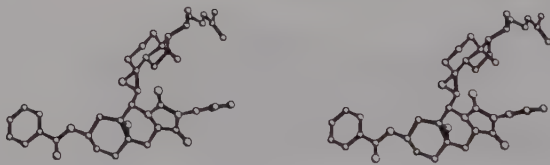


Fig. 1. Stereoscopic view of  $C_{34}H_{48}N_3O_5$ .

SODIUM MONENSINATE B MONOHYDRATE

$C_{35}H_{59}NaO_{11} \cdot H_2O$

Y. BARRANS, M. ALLÉAUME and G. JÉMINET, 1982. *Acta Cryst.*, **B38**, 1144-1149.

Orthorhombic,  $P2_12_12_1$ ,  $a = 12.135$ ,  $b = 14.976$ ,  $c = 20.683$  Å,  $Z = 4$ . Cu radiation,  $R = 0.029$  for 2349 reflexions.

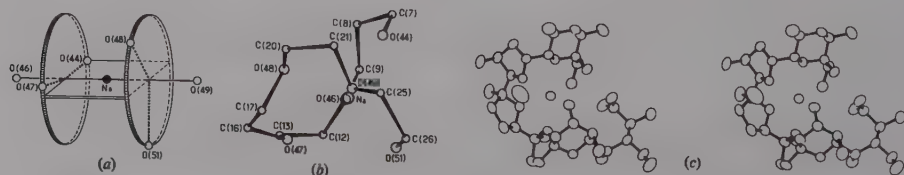


Fig. 1.  $C_{35}H_{59}NaO_{11} \cdot H_2O$ : (a) the oxygen environment of the  $Na^+$  ion, (b) the polyether chain around the  $Na^+$  ion and (c) a stereoscopic view of the structure.

In the complex (Fig. 1) the heterocyclic part of the anion wraps the cation which is coordinated to six O atoms with Na...O distances of 2.33 to 2.47 Å. Two hydrogen bonds between the carboxylate group at one end and the two hydroxyl groups at the other end close the anionic ring. The water molecule is hydrogen-bonded to two anions. The structure is compared with those of other similar complexes.

1'-(2-HYDROXY-5-METHOXY-4-t-BUTYLPHENYL)-4a',5,7'-TRIMETHOXY-4,4',6'-TRI-t-BUTYLSPIRO-(CYCLOHEXA-3,5-DIENE-1,9'-XANTHEN)-2,2'(4'aH)-DIONE CHLOROFORM SOLVATE

$C_{44}H_{56}O_8 \cdot CHCl_3$

L.T. BYRNE, F.R. HEWGILL, F. LEGGE, B.W. SKELTON and A.H. WHITE, 1982. J. Chem. Soc. Perkin I, 2855-2862.

Monoclinic,  $P2_1/n$ ,  $a = 22.48$ ,  $b = 12.401$ ,  $c = 16.461$  Å,  $\beta = 95.14^\circ$ ,  $D_m = 1.20$ ,  $Z = 4$ . Mo radiation,  $R = 0.051$  for 1644 reflexions.

Bond lengths are as expected for the structure (Fig. 1). There is considerable variation in the ring bond angles consistent with the effects of the double bonds. The central heterocyclic ring is distorted into a pseudo-boat. The dihedral angles between its mean plane and the planes of 1a, 2a and 1b are 16.3, 14.8 and  $74.8^\circ$  respectively and the 1a-2a dihedral angle is  $22.8^\circ$ . The asymmetric unit comprises one molecule of the compound and one molecule of chloroform solvent, the hydrogen atom of which is hydrogen bonded to the hydroxyl hydrogen on O(6) of unit 1b  $O(6) \cdots H$  2.14 Å. There is an intermolecular hydrogen bond between H(6) of 2b to O(6) of 1a of 1.7<sub>8</sub> Å.

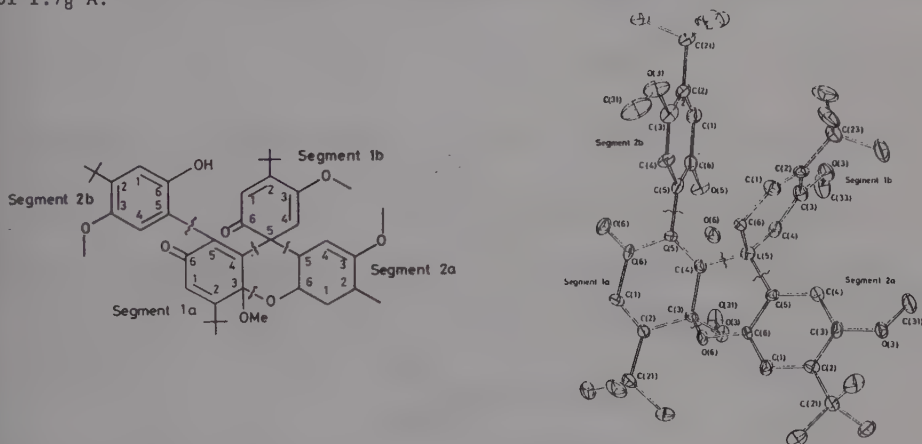


Fig. 1. Molecular skeleton and structure of  $C_{44}H_{56}O_8 \cdot CHCl_3$ .

CYCLO[TETRAKIS[(5-t-BUTYL-2-ACETOXY-1,3-PHENYLENE)METHYLENE]] ACETIC ACID  
 $C_{52}H_{64}O_8 \cdot C_2H_4O_2$

C. RIZZOLI, G.D. ANDRETTI, R. UNGARO and A. POCHINI, 1982. J. Mol. Struct., **82**, 133-141.

Triclinic,  $P\bar{1}$ ,  $a = 13.641$ ,  $b = 13.149$ ,  $c = 14.953$  Å,  $\alpha = 85.03$ ,  $\beta = 73.96$ ,  $\gamma = 93.99^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.085$  for 2721 reflexions.

The configuration of the macrocycle (Fig. 1) is such that three acetoxy groups are oriented on one side with intramolecular contacts forcing two facing phenolic units to be almost parallel. The acetic acid molecules, which are joined in dimers by hydrogen bonding, occupy centrosymmetric cavities in the host structure, whose size of 168 Å<sup>3</sup> is comparable with the van der Waals volume of the dimer of 146 Å<sup>3</sup>.

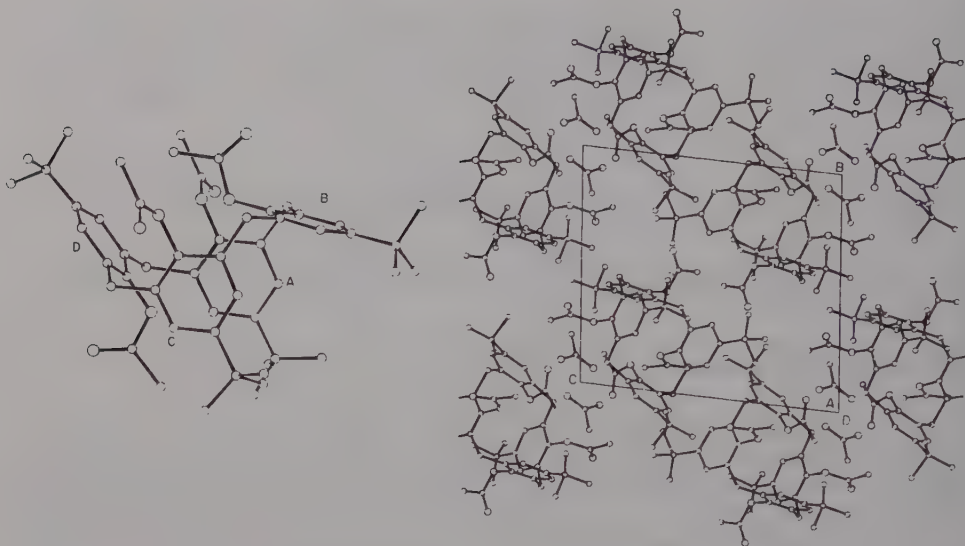


Fig. 1. A view of the tetrameric macrocycle and the crystal packing.

1,3-DITHIETANE-1,1,3,3-TETRAOXIDE



E. BLOCK, E.R. COREY, R.E. PENN, T.L. RENKEN, P.F. SHERWIN, H. BOCK, T. HIRABAYASHI, S. MOHMAND and B. SOLOUKI, 1982. *J. Am. Chem. Soc.*, **104**, 3119-3130.

Monoclinic,  $P2_1/c$ ,  $a = 5.582$ ,  $b = 5.759$ ,  $c = 8.954$  Å,  $\beta = 116.84^\circ$ ,  $D_m = 2.01$ ,  $Z = 2$ . Mo radiation,  $R = 0.032$  for 1138 reflexions.

The centrosymmetric molecule (Fig. 1) contains a planar and nearly square four membered  $\text{S}_2\text{C}_2$  ring (S-C 1.813(2) and 1.804(2) Å). The S-O distances are 1.436(2) and 1.433(2) Å, C-S-C 88.5(1) and S-C-S 91.5(1)°.

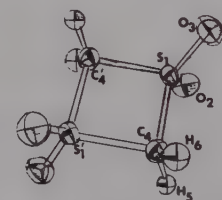
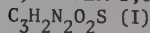
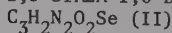


Fig. 1. The 1,3-dithietane-1,1,3,3-tetraoxide molecule.

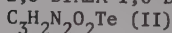
2,5-DIAZA-1,6-DIOXA-6a-THIAPENTALENE



2,5-DIAZA-1,6-DIOXA-6a-SELENAPENTALENE



2,5-DIAZA-1,6-DIOXA-6a-TELLURAPENTALENE



F.A. AMUNDSEN, L.K. HANSEN and A. HORDVIK, 1982. *Acta Chem. Scand.*, **A36**, 673-681.

I. Monoclinic,  $P2_1/c$ ,  $a = 6.939$ ,  $b = 7.000$ ,  $c = 11.013$  Å,  $\beta = 110.83^\circ$ ,  $D_m = 1.71$ ,  $Z = 4$ . Mo radiation,  $R = 0.043$  for 630 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 6.987$ ,  $b = 7.000$ ,  $c = 11.379$  Å,  $\beta = 110.93^\circ$ ,  $D_m = 2.26$ ,  $Z = 4$ . Mo radiation,  $R = 0.061$  for 1127 reflexions.

III. Monoclinic,  $P2_1/c$ ,  $a = 9.605$ ,  $b = 5.006$ ,  $c = 12.202$  Å,  $\beta = 112.94^\circ$ ,  $D_m = 2.74$ ,  $Z = 4$ . Mo radiation,  $R = 0.049$  for 788 reflexions.

Within experimental error, the three molecules are planar and symmetric about the respective C(3a)-X(6a) bonds (Fig. 1). Compounds I and II are isomorphous and III is disordered over two sites, each with 0.5 site occupancy. The average values for bond lengths are: in I, O-N 1.350, C-N 1.309, C-C 1.407 and C-S 1.683 Å; in II, O-N 1.33, C-N 1.33, C-C 1.42 and C-Se 1.827 Å; in III, O-N 1.37, C-N 1.39, C-C 1.36 and C-Te 1.98 Å. These lengths in I and II have been corrected for libration.

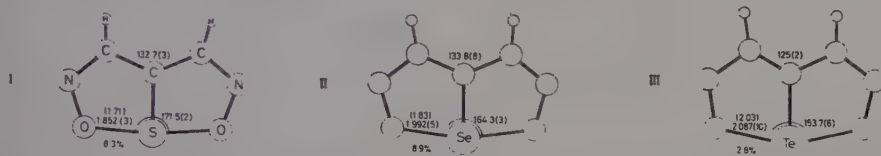


Fig. 1. Molecular structures of  $C_3H_2N_2O_2S$  (I),  $C_3H_2N_2O_2Se$  (II) and  $C_3H_2N_2O_2Te$  (III). The average O-X distances (Å), C-C-C and O-X-O angles ( $^\circ$ ) are given. The corresponding sums (Å) of covalent radii for O and X are shown in square brackets and the relative lengthening of the O-X bonds are given in per cent.

#### HOMOCYSTEINE-THIOLACTONE HYDROCHLORIDE $C_4H_6ClNOS$

G. BOCELLI and M.F. GRENIER-LOUSTALOT, 1982. Cryst. Struct. Comm., **11**, 85-88.

Orthorhombic,  $Pbca$ ,  $a = 19.451$ ,  $b = 9.267$ ,  $c = 7.264$  Å,  $Z = 8$ . Cu radiation,  $R = 0.093$  for 979 reflexions.

The molecule is disordered in the solid state as shown in Fig. 1. The five-membered ring is in envelope conformation with C3 0.60 Å out of the plane of the other four atoms.

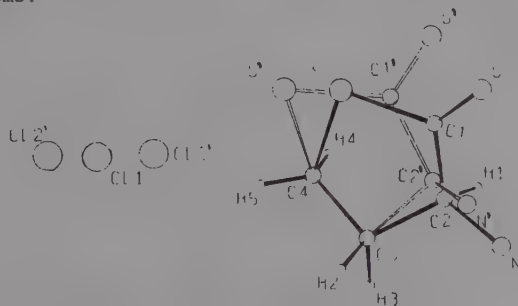


Fig. 1. A view of the disorder in  $C_4H_6ClNOS$ .

#### trans-5-METHYL-cis-5-NITRO-1,3,2-DIOXATHIAN-2-OXIDE $C_4H_7NO_5S$

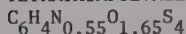
G.H. PETIT, A.T.H. LENSTRA and H.J. GEISE, 1982. Cryst. Struct. Comm., 11, 873-879.  
Orthorhombic,  $Pca2_1$ ,  $a = 13.227$ ,  $b = 5.942$ ,  $c = 18.520$  Å,  $Z = 8$ . Mo radiation,  $R = 0.032$  for 1425 reflexions (at 123 K).

The two molecules in the asymmetric unit have similar ring conformations and differ slightly in nitro group orientation. The conformation found (Fig. 1) was unexpected as it had been anticipated that the  $S=O$  bond would have been axial.



Fig. 1. The ring conformation in  $C_4H_7NO_5S$ .

#### TETRATHIAFULVALENE NITRATE



P. KATHIRGAMANATHAN, M.A. MAZID and D.R. ROSSEINSKY, 1982. J. Chem. Soc. Perkin II, 593-596.

Monoclinic,  $P2_1/n$ ,  $a = 10.976$ ,  $b = 11.868$ ,  $c = 3.607$  Å,  $\beta = 90.79^\circ$ ,  $D_m = 1.69$ ,  $Z = 2$ . Mo radiation,  $R = 0.114$  for 442 reflexions.

This nonstoichiometric salt (Fig. 1) shows unidimensional metallic conducting properties along the  $c$  axis above 291 K. The nitrate ions are completely disordered about the centre of symmetry at 0.5,0,0.5. The S atoms are disordered above and below the plane of the tetrathiafulvalene and this is a novel observation for such compounds and may be involved in the conduction mechanism. The  $C(1)-C(1)$  distance of 1.24 Å is considerably shorter than in tetrathiafulvalene (1.349 Å) but this may in part reflect the effect of the high disorder on the refinement. The crystal structure consists of stacked columns of tetrathiafulvalene and nitrate ions with an interplanar distance of 3.607 Å, close to that reported for similar compounds.

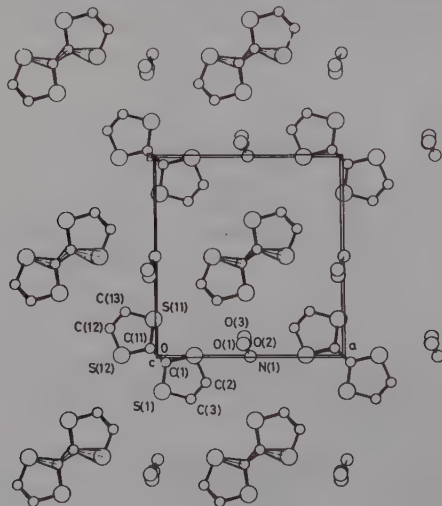
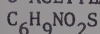


Fig. 1. The crystal structure of  $C_6H_4N_{0.55}O_{1.65}S_4$  viewed along  $c$ .

## 3-ACETYLAMINO-2-THIOLANONE



G. BOCELLI, M.F. GRENIER-LOUSTALOT and P. IRATCABAL, 1982. J. Mol. Struct., 96, 121-126.

Orthorhombic,  $\text{Pna}2_1$ ,  $a = 9.423$ ,  $b = 9.115$ ,  $c = 8.965 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.065$  for 1420 reflexions.

The five-membered ring has an envelope conformation with C(3) 0.56 Å off the plane of the remaining four atoms (Fig. 1). The acetylmino chain is pseudo-equatorial.

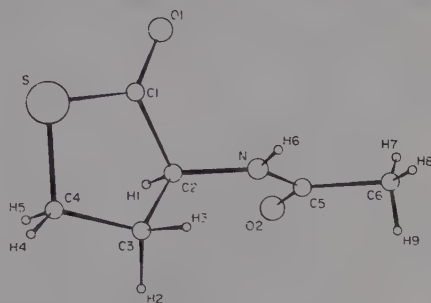
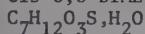


Fig. 1. The  $\text{C}_6\text{H}_9\text{NO}_2\text{S}$  molecule.

## cis-3,3-DIMETHYL-2,4-DIOXA-7-THIABICYCLO[3.3.0]OCTANE 7-OXIDE MONOHYDRATE



F. DAHAN and C. STORA, 1982. Acta Cryst., B38, 1046-1048.

Monoclinic,  $\text{A}2/m$ ,  $a = 6.484$ ,  $b = 8.995$ ,  $c = 15.899 \text{ \AA}$ ,  $\beta = 92.37^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.064$  for 819 reflexions.

The molecule (Fig. 1) is symmetrical with a molecular mirror plane passing through O(S), S, C(3), C(4) and C(5). In the molecule S-C(1) (1.809 Å) and C(1)-C(2) (1.512 Å) are shorter and C(2)-O (1.428 Å) and O-C(3) (1.431 Å) longer than the corresponding bonds in the dimethyl derivative (1). The packing is governed by hydrogen bonds (2.776 Å) between water molecules and O(S) atoms.

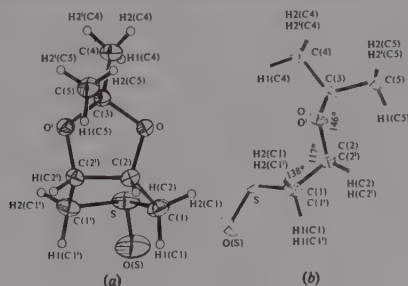


Fig. 1.  $\text{C}_{12}\text{H}_{20}\text{O}_3\text{S}, \text{H}_2\text{O}$ : the molecule viewed along (a) a and (b) b.

1. C. STORA, 1974. C.R. Acad. Sci. Ser. C, 277, 97.



1,3,5,7-TETRAMETHYL-2,4,6,8,9,10-HEXATHIAADAMANTANE ARSENIC TRIIODIDE  
 $C_8H_{12}AsI_3S_6$



R. KNIEP and H.D. RESKI, 1982. *Inorg. Chim. Acta*, **64**, L83-L84.

Monoclinic,  $P2_1/c$ ,  $a = 11.199$ ,  $b = 11.839$ ,  $c = 15.724$  Å,  $\beta = 103.06^\circ$ ,  $D_m = 2.53$ ,  $Z = 4$ . Mo radiation,  $R = 0.078$  for 2713 reflexions.

The As atom bonds to three terminal I atoms in a slightly distorted trigonal pyramidal arrangement with a mean bond distance of 2.576 Å (Fig. 1). Distorted octahedral coordination is achieved by the addition of As-S bonds to three of the six S atoms in the adamantane molecule. The As-S distances range from 3.274 to 3.310 Å and the S-C distances within the adamantane molecule range from 1.791 to 1.847 Å. The angles I-As-I range from  $98.4$  to  $100.3^\circ$ , I-As-S from  $89.5$  to  $111.7$  and  $144.3$  to  $154.7^\circ$ , and S-As-S from  $55.1$  to  $55.2^\circ$ .

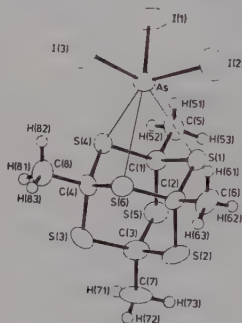


Fig. 1. Molecular structure of  $C_8H_{12}AsI_3S_6$ .

2,6-DINITRATO-9-THIABICYCLO[3.3.1]NONANE 9,9-DIOXIDE  
 $C_8H_{12}N_2O_8S$

P.H. McCABE and G.A. SIM, 1982. *J. Chem. Soc. Perkin II*, 819-821.

Monoclinic,  $A2/a$ ,  $a = 15.144$ ,  $b = 7.851$ ,  $c = 19.958$  Å,  $\beta = 90.295^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.040$  for 2718 reflexions.

The C-C-C angles within the six membered rings are increased beyond the tetrahedral angle, the angles at C(4) and C(8) (Fig. 1) being largest ( $117.5$  and  $116.9^\circ$ ), while those at C(2), C(6), C(3) and C(7) are  $115.4$ ,  $114.8$ ,  $114.1$  and  $113.3^\circ$  respectively. The S-C-C angles at C(1) and C(5) are smaller, ranging from  $106.0$  to  $108.8$  (mean  $107.4^\circ$ ). The nitrate groups cause skewing of the six membered rings, reducing the symmetry of the bicyclo[3.3.1]nonane from  $C_{2v}$  to approximately  $C_2$ . However the positions of the nitrate groups do not conform to  $C_2$  symmetry, with the torsion angles  $N(1)-O(3)-C(2)-C(1)$  and  $N(2)-O(6)-C(6)-C(5)$   $-85.5$  and  $-156.3^\circ$  respectively. The C(3)...C(7) separation of  $3.128$  Å is comparable with that observed in other bicyclo[3.3.1]nonanes.

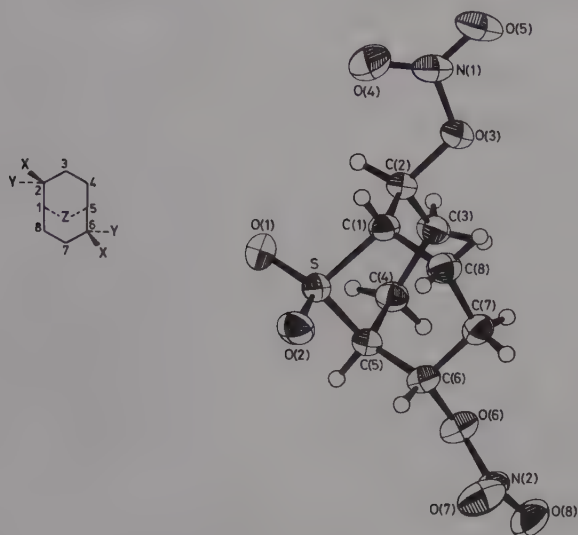
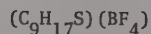
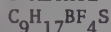


Fig. 1. Molecular skeleton ( $x = \text{ONO}_2$ ,  $y = \text{H}$ ,  $z = \text{SO}_2$ ) and structure of  $\text{C}_8\text{H}_{12}\text{N}_2\text{O}_8\text{S}$ .

8-METHYL-8-THIONIABICYCLO[4.3.0]NONANE TETRAFLUOROBORATE



G.D. ANDRETTI, F. BERNARDI, A. BOTTONI and A. FAVA, 1982. J. Am. Chem. Soc., 104, 2176-2183.

Monoclinic,  $\text{C2/c}$ ,  $a = 19.489$ ,  $b = 12.103$ ,  $c = 11.888 \text{ \AA}$ ,  $\beta = 120.36^\circ$ ,  $D_m = 1.35$ ,  $Z = 8$ . Cu radiation,  $R = 0.072$  for 1584 reflexions.

The structure consists of a heterocyclic sulphonium cation and a  $\text{BF}_4^-$  anion (Fig. 1). The S-C distances are 1.813(6), 1.815(6) and 1.672(7)  $\text{\AA}$  (the latter shortened by thermal motion, as are the B-F bonds of the anion). Other geometrical parameters are normal.

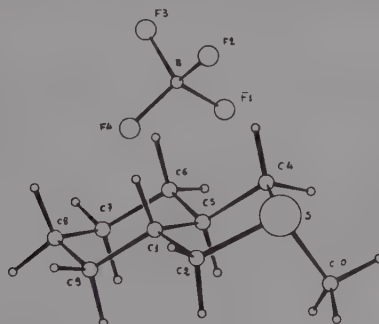


Fig. 1. View of  $(\text{C}_9\text{H}_{17}\text{S})(\text{BF}_4)$ .

## HEXACHLORONAPHTHALENE-1,8-DISULPHIDE



J.H. NOORDIK and M.R. KLEIJBURG, 1982. *Cryst. Struct. Comm.*, **11**, 1335-1338.

Monoclinic, Pn,  $a = 10.68$ ,  $b = 3.853$ ,  $c = 15.31$  Å,  $\beta = 93.3^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.098$  for 460 reflexions.

The molecule (Fig. 1) is planar to within 0.13 Å. The S-S distance is 2.49(2) Å.

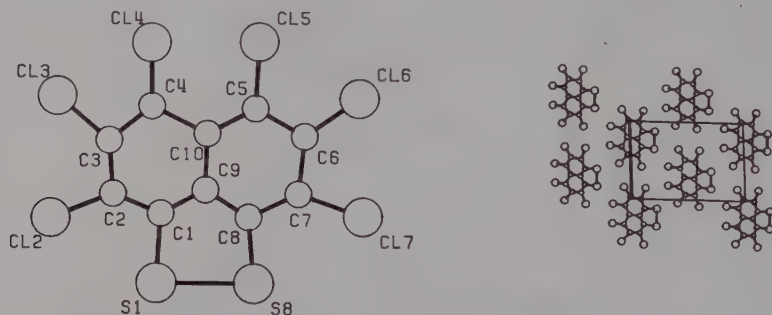
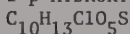


Fig. 1. The  $\text{C}_{10}\text{Cl}_6\text{S}_2$  molecule and packing.

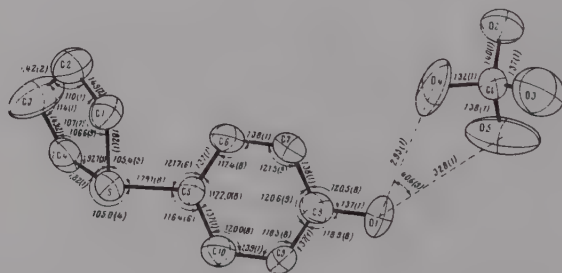
## 1-p-HYDROXYPHENYLTHIOPHANUM PERCHLORATE



D.S. YUFIT, A.I. YANOVSKII, A.E. KALININ, Yu.T. STRUCHKOV, L.R. BARYKINA and E.N. KARAULOVA, 1982. *Kristallografiya*, **27**, 258-261 [*Sov. Phys. Crystallogr.*, **27**, 158-160].

Monoclinic,  $P2_1/c$ ,  $a = 7.987$ ,  $b = 16.505$ ,  $c = 9.346$  Å,  $\beta = 91.12^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.090$  for 1097 reflexions.

The structure consists of ion pairs (Fig. 1) linked by a bifurcated O-H...O hydrogen bond. The S atom in the thiophanium ring has a pyramidal bond configuration and the conformation of the ring is a flattened envelope with C(3) lying 0.38 Å from the plane of the other four atoms. The dihedral angle between the mean planes of the five- and six-membered rings is  $80^\circ$ .



## 3-t-BUTYL-3-(2-THIOPHENO)PROPIONIC ACID MONOHYDRATE



C. REISS, H. SCHENK and C.H. STAM, 1982. Cryst. Struct. Comm., **11**, 2025-2028.

Orthorhombic,  $P2_12_12_1$ ,  $a = 6.0122$ ,  $b = 13.839$ ,  $c = 15.292 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.078$  for 591 reflexions.

The molecule is depicted in Fig. 1. The chain C(7)C(6)C(5)C(8)C(9) is roughly planar, the largest deviation from its best plane being  $0.15 \text{ \AA}$ . The carboxyl group and the thiophene ring make angles of  $54$  and  $80^\circ$  with this plane respectively. The water molecules form hydrogen bonds with the carboxyl O atoms, interconnecting the molecules to form double strings around the screw axes parallel to  $[100]$ . Each water molecule forms three hydrogen bonds, one acceptor bond with O(1) of length  $2.58(2) \text{ \AA}$  and two donor bonds with O(2) of lengths  $2.76(2)$  and  $2.77(2) \text{ \AA}$ .

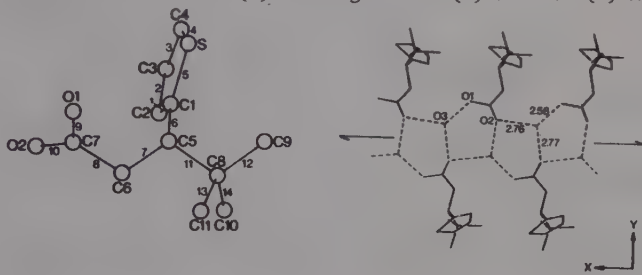
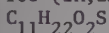


Fig. 1. The  $\text{C}_{11}\text{H}_{16}\text{O}_2\text{S}$  molecule and details of hydrogen bonding.

## rel-(1R,2S,3R,4R)-3-HEXYL-2-HYDROXYMETHYL-4-METHYLTHIETAN 1-OXIDE



D.N. JONES, T.P. KOGAN, P. MURRAY-RUST, J. MURRAY-RUST and R.F. NEWTON, 1982. J. Chem. Soc. Perkin I, 1325-1332.

Monoclinic,  $P2_1/c$ ,  $a = 12.19$ ,  $b = 9.16$ ,  $c = 11.62 \text{ \AA}$ ,  $\beta = 97.16^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.081$  for 1002 reflexions [crystals began to decompose part-way through data collection].

The predicted configuration of the thietan prostaglandin analogue (Fig. 1) is confirmed by the X-ray structure. The thietan ring is folded about the two  $\alpha$ -C atoms by  $151^\circ$ . The hexyl substituent has an extended conformation except for a synclinal arrangement about C(9)-C(10). The molecules exist as hydrogen-bonded enantiomeric pairs, joined by hydrogen bonds between the hydroxyl oxygen of one molecule and the sulphanyl oxygen of the other. The H atoms were not located, but the predicted intermolecular hydrogen bond is  $2.71 \text{ \AA}$  compared with the intramolecular distance of  $3.65 \text{ \AA}$  which is too long for hydrogen bonding.

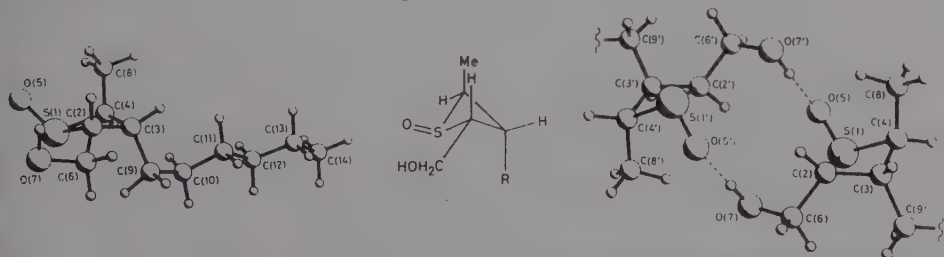


Fig. 1. Molecular structure, molecular skeleton and intermolecular hydrogen bonding in  $\text{C}_{11}\text{H}_{22}\text{O}_2\text{S}$ .

## AZULENO[1,2-b]THIOPHENE

 $C_{12}H_8S$ S. KASHINO, M. HAISA, K. FUJIMORI and K. YAMANE, 1982. Acta Cryst., **B38**, 2729-2731.Orthorhombic,  $Pnma$ ,  $a = 7.954$ ,  $b = 19.194$ ,  $c = 6.021$  Å,  $D_m = 1.33$ ,  $Z = 4$ . Cu radiation,  $R = 0.105$  for 504 reflexions.

In the crystal structure (Fig. 1) the molecules form a sheet parallel to (010). The sheets are stacked along  $b$ . The structure is disordered as in azulene. The molecule is planar within  $0.06(2)$  Å. The bond-length alternation in the seven-membered ring is significant, the shortest C-C length being  $1.27(2)$  Å between positions 5 and 6 (C(7)-C(8)), and the longest  $1.58(2)$  Å between 4a and 9a (C(11)-C(12)).

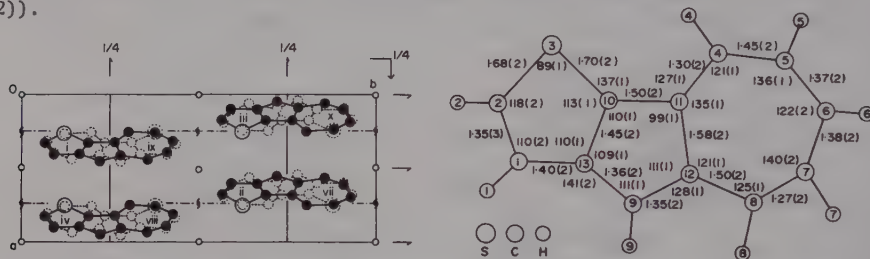


Fig. 1.  $C_{12}H_8S$ : bond lengths and angles in the molecule and the projection of the crystal structure along  $c$ ; broken lines indicate the disordered molecules.

## 10-(1,3-DITHIOLAN-2-YLIDENE)-10H-INDENO[1,2-f]-1,2,3,4,5-PENTATHIEPIN

 $C_{12}H_8S_7$ J.D. KÖRP, I. BERNAL, S.F. WATKINS and F.R. FRONCZEK, 1982. J. Heterocyclic Chem., **19**, 459-462.Triclinic,  $P\bar{1}$ ,  $a = 8.931$ ,  $b = 9.387$ ,  $c = 10.175$  Å,  $\alpha = 75.73$ ,  $\beta = 73.35$ ,  $\gamma = 64.37^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.033$  for 1925 reflexions.

The molecule consists of an indene core with a nearly co-planar dithiolane and a fused pentasulfide chain. The  $S_5C_2$  ring is in the chair configuration, with an average S-S distance of  $2.052$  Å. There is no variation of bond lengths as is frequently seen in multi-sulfur chains. The indenone ring shows no evidence of any delocalization, while the dithiolane ring is disordered at the two methylene positions. The S-C bond lengths in the  $C_2S_5$  ring average  $1.762$  Å.

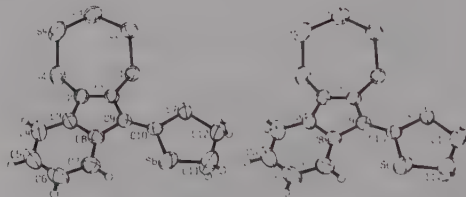


Fig. 1. The  $C_{12}H_8S_7$  molecule (stereo pair).

## 8a-METHYL-1β-ACETOXY-3-THIA-6-DECALONE

 $C_{12}H_{18}O_3S$ H. KOYAMA and Y. YOKOUCHI, 1982. Cryst. Struct. Comm., **11**, 1959-1964.

Monoclinic,  $P2_1/c$ ,  $a = 10.695$ ,  $b = 9.355$ ,  $c = 17.545$  Å,  $\beta = 134.75^\circ$ ,  $D_m = 1.282$ ,  $Z = 4$ . Mo radiation,  $R = 0.072$  for 2149 reflexions.

The two six-membered rings have chair form (Fig. 1) with a trans ring junction. Molecular dimensions are as anticipated (mean C-S 1.810(6) Å).

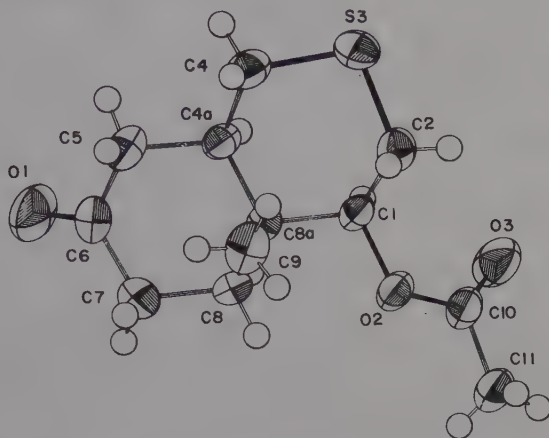


Fig. 1. A view of  $C_{12}H_{18}O_3S$ .

5-ETHOXY-4-METHYL-3-(4-TOLYLSULPHONYLIMINO)-3H-DITHIOLE

$C_{13}H_{15}NO_3S_3$  (I)

6-ETHYLTHIO-1,3-DIPHENYL-2-THIOURACIL

$C_{18}H_{16}N_2OS_2$  (II)

E. SCHAUMANN, H.-G. BAUCH, S. SIEVEKING and G. ADIWIDJAJA, 1982. Chem. Ber., **115**, 3340-3352.

I. Monoclinic,  $C2/c$ ,  $a = 27.836$ ,  $b = 7.521$ ,  $c = 15.727$  Å,  $\beta = 113.50^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.040$  for 1843 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 12.559$ ,  $b = 5.807$ ,  $c = 23.216$  Å,  $\beta = 94.89^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.10$  for 1003 reflexions.

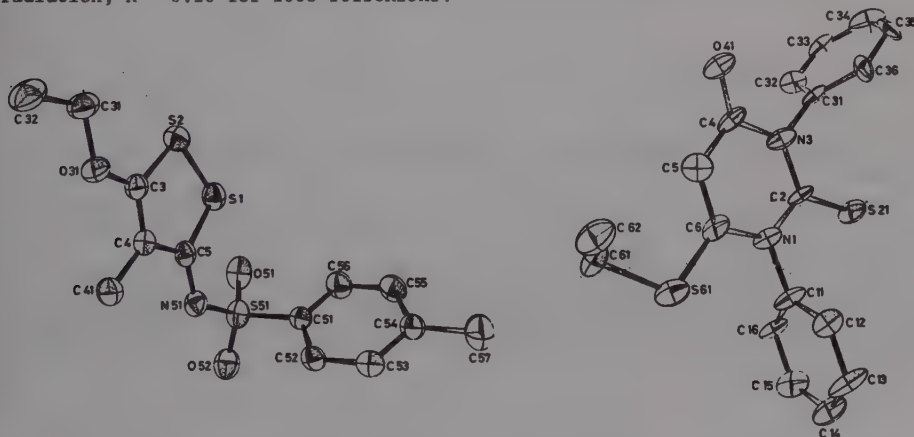


Fig. 1. The structures of  $C_{13}H_{15}NO_3S_3$  (I) (left) and  $C_{18}H_{16}N_2OS_2$  (II) (right).



The structures of I and II were confirmed as those shown in Fig. 1 with normal values for bond lengths and angles.

ANTHRA[9,1-cd:10,5-c'd']BIS[1,2]DISELENOLE (TETRASELENAANTHRACENE)  
 $C_{14}H_6Se_4$  (I)

TETRASELENAANTHRACENIUM IODIDE  
 $C_{14}H_6I_{1.20}Se_4$  (II)

H. ENDRES, H.J. KELLER, J. QUECKBÖRNER, D. SCHWEITZER and J. VEIGEL, 1982. Acta Cryst., B38, 2855-2860.

I. Monoclinic,  $P2_1/n$ ,  $a = 9.318$ ,  $b = 4.111$ ,  $c = 16.035$  Å,  $\beta = 90.43^\circ$ ,  $Z = 2$ .  
 Mo radiation,  $R = 0.030$  for 1039 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 18.644$ ,  $b = 3.856$ ,  $c = 19.746$  Å,  $\beta = 93.36^\circ$ ,  $Z = 4$ .  
 Mo radiation,  $R = 0.118$  for 1080 reflexions.

In compound I the planar molecules form stacks along  $b$  with an interplanar separation of 3.59 Å (Fig. 1). The normals to the molecular planes are inclined at  $29^\circ$  to the stacking axis. Short intermolecular Se...Se contacts exist between adjacent stacks, the shortest one being 3.467(1) Å. The structure of compound II contains two crystallographically inequivalent stacks of partially oxidised 'tetraselelenaanthracenium' cations (interplanar distances 3.44 and 3.56 Å respectively) and linear triiodide chains ordered in one dimension only (Fig. 1).

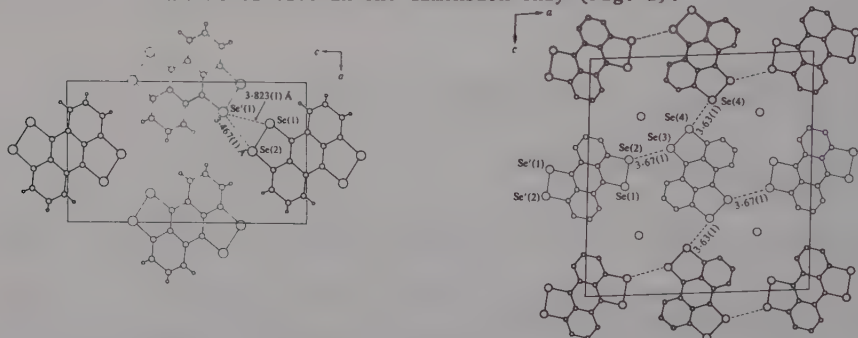


Fig. 1.  $C_{14}H_6Se_4$  (I) and  $C_{14}H_6I_{1.20}Se_4$  (II): projections of the structures of I and II on to the  $ac$  planes.

$\Delta^{2,2'}$ -BI-d-BENZO-1,3-DITHIOL  
 $C_{14}H_8S_4$

R.P. SHIBAIEVA, R.M. LOBKOVSKAYA and V.N. KLYUEV, 1982. Cryst. Struct. Comm., 11, 835-839.

Monoclinic,  $P2_1/b$ ,  $a = 12.093$ ,  $b = 14.580$ ,  $c = 3.948$  Å,  $\gamma = 114.27^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.034$  for 1037 reflexions.

The molecules (Fig. 1) lie on inversion centres and have a mean interplanar stack distance along  $c$  of 3.544 Å. The molecule has a 'flat-chair' conformation with a  $4^\circ$  angle between the plane of the central six atom  $C_2S_4$  moiety and the two-fused-ring system.

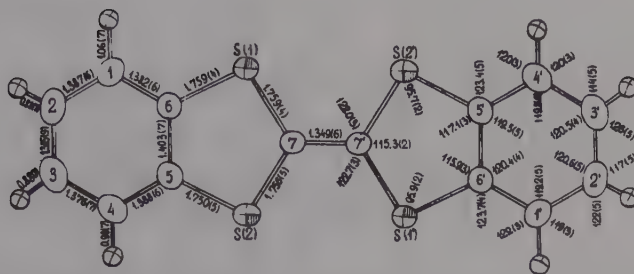
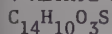


Fig. 1. Bond lengths and angles in  $C_{14}H_8S_4$ .

#### 4-METHYL-9-OXOTHIOXANTHENE 10,10-DIOXIDE



J. LONGO and M.F. RICHARDSON, 1982. *Acta Cryst.*, **B38**, 2724-2726.

Orthorhombic,  $Pnma$ ,  $a = 14.384$ ,  $b = 6.882$ ,  $c = 11.705$  Å,  $D_m = 1.48$ ,  $Z = 4$ . Mo radiation,  $R = 0.058$  for 776 reflexions.

In the molecule (Fig. 1) the thioxanthen-9-one ring system is strictly planar, as all of the ring atoms are required to lie on crystallographic mirror planes. Bond lengths and angles are comparable to those observed in similar structures: mean C-S 1.767(2) Å, C-S-C 106.4(3)°, S-O(2) 1.431(4) Å. The molecules are linked into chains through weak C-H...O hydrogen bonds. The overall packing arrangement is a layered one, but the ring systems in one layer do not stack above the ring systems in an adjoining layer.

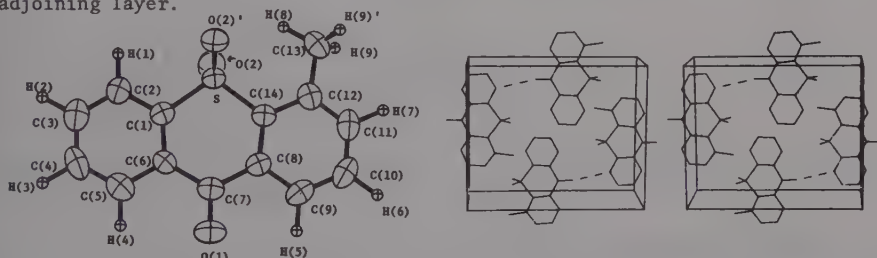
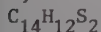


Fig. 1.  $C_{14}H_{10}O_3S$ : a perspective view of the molecule and a stereoscopic view of the unit-cell contents;  $a$  is horizontal,  $c$  is vertical.

#### 2,7-DIMETHYLTHIANTHRENE



T.J.R. WEAKLEY, 1982. *Cryst. Struct. Comm.*, **11**, 681-684.

Orthorhombic,  $P2_12_12_1$ ,  $a = 11.367$ ,  $b = 13.202$ ,  $c = 8.180$  Å,  $Z = 4$ . Cu radiation,  $R = 0.048$  for 853 reflexions.

The molecule has non-crystallographic twofold symmetry (Fig. 1). The angle between the planes of the aromatic rings is 137.0° and the central ring has a boat conformation with C-S 1.760-1.774(7) Å.

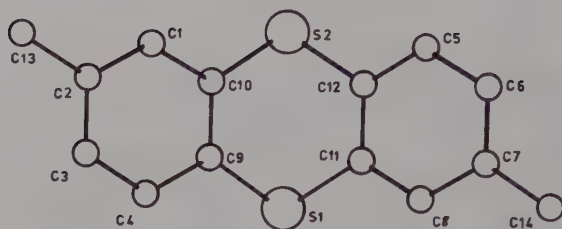


Fig. 1. A view of 2,7-dimethylthianthrene; the approximate twofold symmetry axis is normal to the S...S vector.

3a,6,7,8,9,9a-HEXAHYDRO-3a,5-DIMETHYL-THIENO[3,2-b][2]BENZOTHIOPHENE-2,3-DICARBONITRILE

$C_{14}H_{14}N_2S_2$

R.H. HALL, H.J. DEN HERTOOG, Jr., D.N. REINHOUT, S. HARKEMA, G.J. VAN HUMMEL and J.W.H.M. UITERWIJK, 1982. *J. Org. Chem.*, **47**, 977-982.

Triclinic,  $P\bar{1}$ ,  $a = 8.743$ ,  $b = 11.369$ ,  $c = 7.714$  Å,  $\alpha = 94.29$ ,  $\beta = 104.70$ ,  $\gamma = 108.90^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.035$  for 1945 reflexions.

The analysis establishes the structure as shown in Fig. 1.

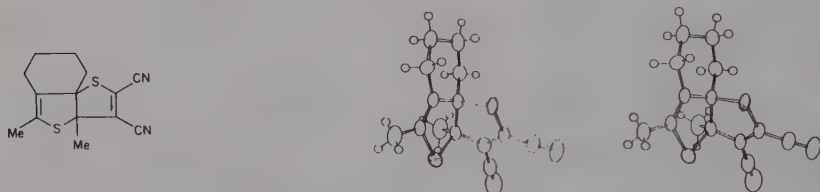


Fig. 1. The  $C_{14}H_{14}N_2S_2$  molecule and a stereoview.

7,16-DITHIADISPIRO[5.2.5.2]HEXADECANE

$C_{14}H_{24}S_2$

K. STADNICKA, L. LEBIODA and K. SUWIŃSKA, 1982. *Acta Cryst.*, **B38**, 985-988.

Monoclinic,  $P2_1/n$ ,  $a = 17.912$ ,  $b = 6.188$ ,  $c = 12.385$  Å,  $\beta = 95.14^\circ$ ,  $D_m = 1.24$ ,  $Z = 4$ . Mo radiation,  $R = 0.048$  for 1908 reflexions.

The structure (Fig. 1) contains disordered enantiomeric molecules with site-occupancy factors of about 0.9 and 0.1 respectively. Both cyclohexane rings and the dithiane moiety have chair conformations.

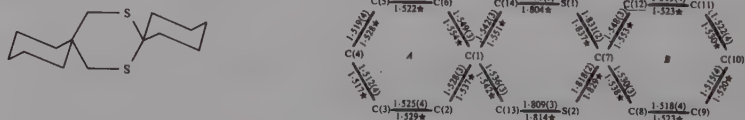
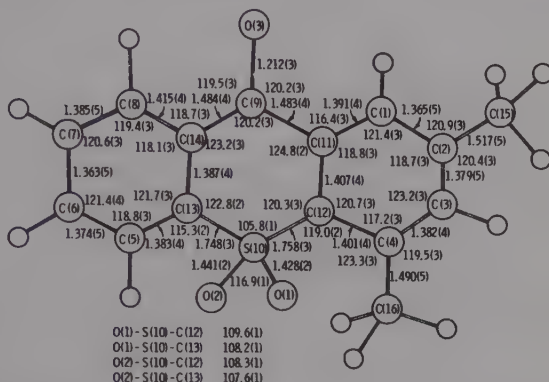
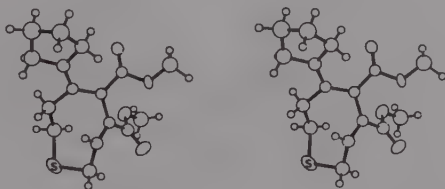


Fig. 1.  $C_{14}H_{24}S_2$ : bond lengths in the major component (values marked with an asterisk are corrected for thermal vibration).

$$\text{C}_{15}\text{H}_{12}\text{O}_3\text{S}$$

$$\text{C}_{15}\text{H}_{21}\text{NO}_4\text{S}$$

$$\text{C}_{16}\text{H}_{12}\text{O}_2\text{S}$$

P. GENESTE, A. GUIDA, D. LEVACHÉ, P. BRIARD, R. ROQUES, J.P. DECLERCQ and G. GERMAIN, 1982. *Cryst. Struct. Comm.*, **11**, 227-230.

Monoclinic,  $P2_1/c$ ,  $a = 11.600$ ,  $b = 10.998$ ,  $c = 11.094 \text{ \AA}$ ,  $\beta = 116.82^\circ$ ,  $Z = 4$ . Mo radiation,  $R = [?]$  for 1408 reflexions.

The molecule (Fig. 1) appears as formed of two folded parts in two different planes. The first is the benzo(b)thiophene system and the second is formed by the two other rings with an angle in between of  $103.5^\circ$ . The dihedral angle  $H_{6a}C_{6a}C_{11b}H_{11b}$  at the junction is equal to  $32^\circ$ , and the cyclohexadiene and the thiophene rings are in half chair conformations.

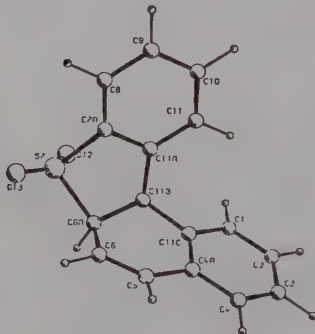


Fig. 1. The  $C_{16}H_{12}O_2S$  molecule.

5-NITROSO-4-PHENETHYLBENZO[b]THIOPHENE  
 $C_{16}H_{13}NOS$

K. PROUT and F.M. MIAO, 1982. *Acta Cryst.*, B38, 685-687.

Monoclinic,  $C2/c$ ,  $a = 17.313$ ,  $b = 10.09$ ,  $c = 15.463 \text{ \AA}$ ,  $\beta = 103.12^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.039$  for 940 reflexions.

The asymmetric unit contains one molecule of the nitroso monomer (Fig. 1). There are no weak bonding interactions involving the nitroso group. Some dimensions are,  $N(10)-O(11)$  1.213(5) - the shortest reported for a C-nitroso compound -  $C(6)-N(10)$  1.419(5)  $\text{\AA}$  and  $C(5)-C(6)-C(7)$   $123.7(5)^\circ$ .

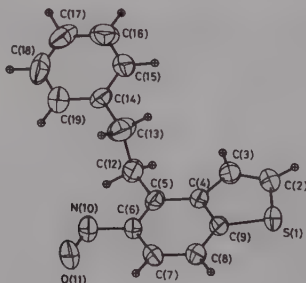


Fig. 1. The 5-nitroso-4-phenethylbenzo[b]thiophene

2,3,7,8-TETRAMETHOXYTHIANTHRENE  
 $C_{16}H_{16}O_4S_2$

W. HINRICHS, H.-J. RIEDEL and G. KLAR, 1982. *J. Chem. Research*, S, 334-335; M, 3501-3539.

Monoclinic,  $P2_1/n$ ,  $a = 12.487$ ,  $b = 30.586$ ,  $c = 12.637$  Å,  $\beta = 95.56^\circ$ ,  $Z = 12$ . Mo radiation,  $R = 0.065$  for 3635 reflexions.

The asymmetric unit contains three molecules, two being stacked above each other, the third one lying perpendicular to this pair of molecules. In the crystal the asymmetric units are arranged in such a way that no further stacks are formed. The molecule (Fig. 1) is folded along the S...S axis (fold angle  $131^\circ$ ) in a 'butterfly' conformation (C-S 1.776 Å, C-S-C  $100.2^\circ$ ). The methoxy substituents are coplanar with their phenyl rings.

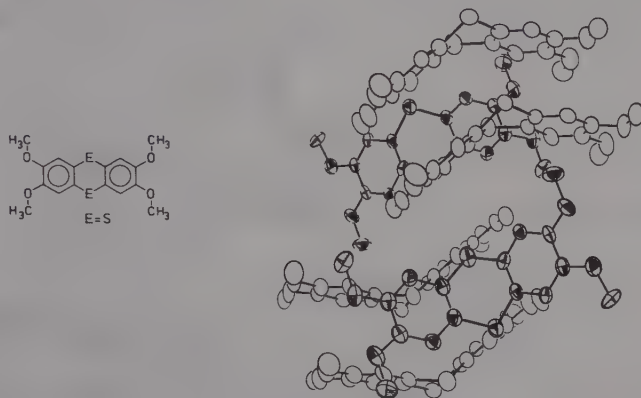


Fig. 1.  $C_{16}H_{16}O_4S_2$ : the molecular skeleton and part of the crystal structure.

syn-2,11-DISELENA[3,3]METACYCLOPHANE  
 $C_{16}H_{16}Se_2$

G.W. BUSHNELL and R.H. MITCHELL, 1982. *Canad. J. Chem.*, **60**, 362-367.

Monoclinic,  $P2_1/n$ ,  $a = 19.26$ ,  $b = 8.014$ ,  $c = 9.24$  Å,  $\beta = 98.9^\circ$ ,  $D_m = 1.733$ ,  $Z = 4$ . Mo radiation,  $R = 0.039$  for 2044 reflexions.

The molecule (Fig. 1) has the syn-conformation with the largest possible Se...Se separation (7.218 Å). The benzenoid rings form a dihedral angle of  $19.1^\circ$ . The mean Se-C distance is 1.964(5) Å and mean C-Se-C  $100.9(2)^\circ$ .

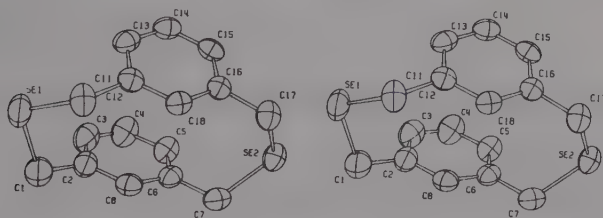


Fig. 1. A stereoview of  $C_{16}H_{16}Se_2$ .

2-(N-ACETYL-N-3-NITROPHENYLAMINOMETHYLENE)-3-(2H)BENZO[b]THIOPHENONE  
 $C_{17}H_{12}N_2O_4S$  (I)

N-(3-NITROPHENYL)-3-ACETOXY-2-BENZO[b]THIOPHENALDIMINE  
 $C_{17}H_{12}N_2O_4S$  (II)



S.M. ALDOSHIN, O.A. DYACHENKO, L.O. ATOVMYAN, V.I. MINKIN, V.A. BREN and G.D. PALUY, 1982. *Z. Krist.*, 159, 143-159.

I. Monoclinic,  $P2_1/c$ ,  $a = 14.503$ ,  $b = 14.459$ ,  $c = 7.666$  Å,  $\beta = 99.52^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.045$  for 955 reflexions.

II. Orthorhombic,  $Fdd2$ ,  $a = 28.950$ ,  $b = 41.552$ ,  $c = 5.099$  Å,  $Z = 16$ . Cu radiation,  $R = 0.043$  for 580 reflexions.

The compounds are photoisomers. I has a ketoamine structure with Z-configuration at C(1)-C(9) (Fig. 1). II has an enolamine structure with the acyl group on O(1).

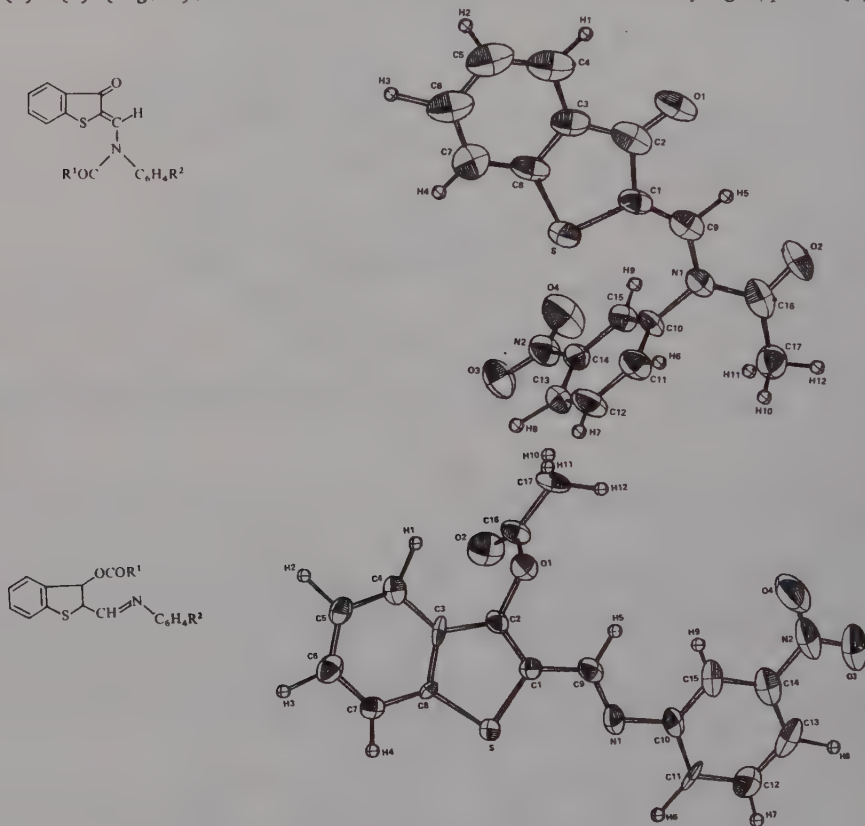


Fig. 1. The two isomers of  $C_{17}H_{12}N_2O_4S$ , I (top) and II (bottom).

trans-9-ETHYL-2,4-DIMETHYLTHIOXANTHENE 10-OXIDE  
 $C_{17}H_{18}OS$

S.S.C. CHU and V. NAPOLEONE, 1982. *Acta Cryst.*, B38, 2288-2290.

Triclinic,  $P\bar{1}$ ,  $a = 8.019$ ,  $b = 8.615$ ,  $c = 11.144$  Å,  $\alpha = 104.09$ ,  $\beta = 77.30$ ,  $\gamma = 103.75^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.049$  for 2121 reflexions.

In the molecule (Fig. 1) the 9-ethyl group is in the 'boat-axial' and the 10-oxide group in the 'boat-equatorial' conformation with respect to the central ring of the thioxanthene ring system. There are no intermolecular contacts less than van der Waals distances.

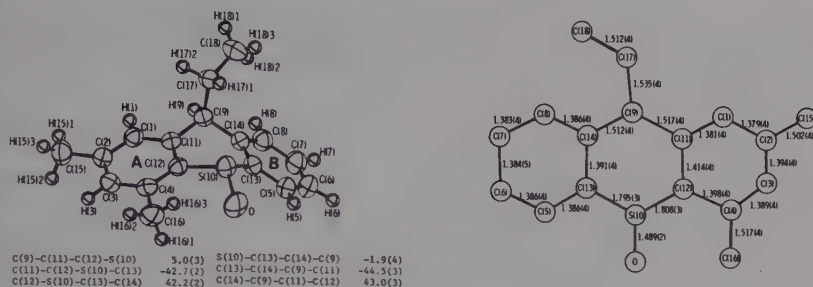


Fig. 1.  $C_{17}H_{18}OS$ : a perspective view of the molecule and bond lengths in the molecule.

3,4-DIHYDRO-2H-[1]BENZOTHIENO[3,2-b]PYRAN-2-SPIRO-2' (3'H)-(1-BENZOTHIOPHEN)-3'-ONE  
 $C_{18}H_{12}O_2S_2$

P.J. COX and R.A. HOWIE, 1982. Acta Cryst., B38, 657-659.

Monoclinic,  $P2_1/c$ ,  $a = 9.722$ ,  $b = 12.982$ ,  $c = 11.991$  Å,  $\beta = 99.40^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.050$  for 1695 reflexions.

In this spiro-fused dimer (Fig. 1) the benzothiophene moieties are almost planar with a dihedral angle between the planes of  $83^\circ$ . The dihydropyran ring has a half-chair conformation. The spiro planes  $S(2)C(4)C(12)$  and  $C(3)C(4)O(1)$  are inclined at  $92.3^\circ$ . Some bond lengths are,  $C(4)-C(12)$  1.556(7),  $S-C(sp^2)$  1.733-1.743,  $S-C(sp^2)$  1.847 Å.

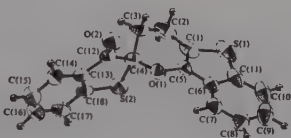


Fig. 1. The  $C_{18}H_{12}O_2S_2$  molecule.

2-[3-(o-NITROPHENYLTHIO)-1-INDENYLIDENE]-1,3-DITHIOLANE  
 $C_{18}H_{13}NO_2S_3$  (I)

2-[3-(o-NITROPHENYLTHIO)-1-INDENYLIDENE]-1,3-DITHIOLE  
 $C_{18}H_{11}NO_2S_3$  (II)

2-{3-[1-(1,3-DITHIOLAN-2-YLIDENE)-2,3-DIHYDRO-2-INDENYL]-1-INDENYLIDENE}-1,3-DITHIOLANE  
 $C_{24}H_{20}S_4$  (III)

J.C. BARNES, J.D. PATON and B.H. NICHOLLS, 1982. Acta Cryst., B38, 1525-1529.

I. Monoclinic,  $P2_1/c$ ,  $a = 11.584$ ,  $b = 10.935$ ,  $c = 16.173$  Å,  $\beta = 121.5^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.047$  for 1852 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 10.782$ ,  $b = 8.40$ ,  $c = 10.501$  Å,  $\alpha = 107.95^\circ$ ,  $\beta = 69.23^\circ$ ,  $\gamma = 109.0^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.069$  for 1486 reflexions.

III. Monoclinic,  $P2_1/n$ ,  $a = 12.23$ ,  $b = 7.58$ ,  $c = 23.43$  Å,  $\beta = 105.96^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.057$  for 1600 reflexions.

In each molecule (Fig. 1) the five-membered dithio ring is twisted slightly (mean  $7.4^\circ$ ) out of the indene plane reducing the contacts  $S(11)\dots C(7)$ ,  $S(11)\dots C(8)$ ,

S(14)...C(2) to 3.29, 3.28, 3.11 Å respectively. The nitrophenylthio group in I and II and the second indene group in III are twisted  $72 \pm 5^\circ$  from the indene plane. The nitro group is  $10.9(7)$  and  $24.6(5)^\circ$  out of the phenyl plane in I and II. Molecular geometry is normal.

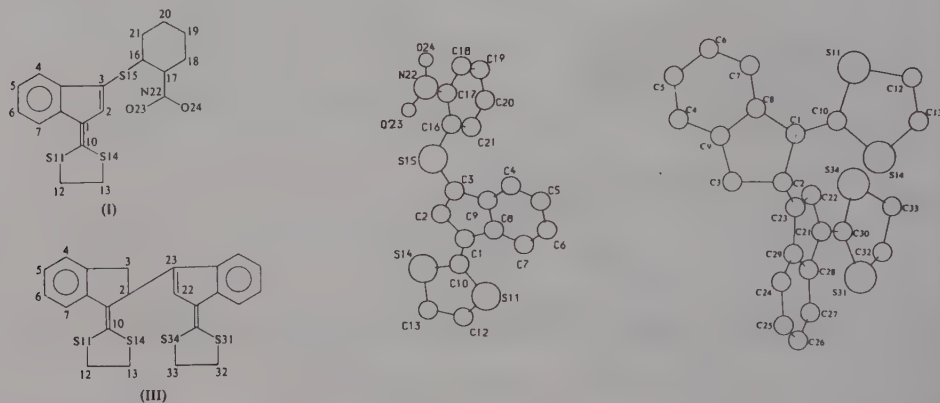


Fig. 1. The  $C_{18}H_{13}NO_2S_3$  (I) and  $C_{24}H_{20}S_4$  (III) molecules, and views of (I) centre and (III) right.

N,N'-BIS[2-(5-t-BUTYL-3H-1,2-DITHIOL-3-YLIDENE)ETHYLIDENE]HYDRAZINE  
 $C_{18}H_{24}N_2S_4$

A.F. CUTHBERTSON, C. GLIDEWELL and D.C. LILES, 1982. Acta Cryst., B38, 2281-2283.

Triclinic,  $P\bar{1}$ ,  $a = 5.929$ ,  $b = 8.898$ ,  $c = 9.954$  Å,  $\alpha = 92.05$ ,  $\beta = 102.12$ ,  $\gamma = 104.73^\circ$ ,  $Z = 1$ . Mo radiation,  $R = 0.0959$  for 1407 reflexions.

The molecule (Fig. 1) lies across a centre of inversion and is planar apart from the t-butyl groups. The S(1)-S(2) and S(1)...N(1) distances are, respectively, 2.124(3) and 2.489(6) Å. The molecules are stacked in the crystal with an inter-ring spacing of 4.61(2) Å.

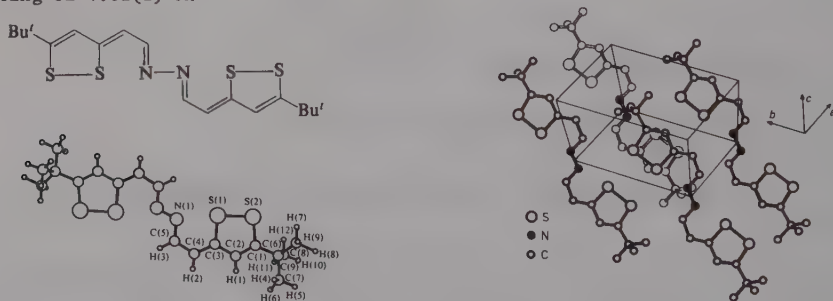


Fig. 1.  $C_{18}H_{24}N_2S_4$ : perspective views of the molecule and of the unit cell.

2-(N-PHENYL-N-DITRIFLUOROMETHYLACETYLAMINOMETHYLENE)-3(2H)-BENZO[b]THIOPHENONE  
 $C_{19}H_{11}F_6NO_2S$

S.M. ALDOSHIN, O.A. D'YACHENKO, L.O. ATOVMYAN, V.I. MINKIN, V.A. BREN' and G.D. PALUI, 1982. Zh. Strukt. Khim., 23-4, 107-110 [J. Struct. Chem., 23, 579-582].

Monoclinic,  $P2_1/b$ ,  $a = 12.771$ ,  $b = 7.759$ ,  $c = 19.154$  Å,  $\gamma = 104.3^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.076$  for 1124 reflexions.

The molecule (Fig. 1) has a ketoamine structure with a  $Z$  configuration relative to the  $C_1=C_9$  bond. Bond lengths and angles ( $\sigma = 0.005$ - $0.010$  Å and  $0.3$ - $1.0^\circ$ ) are normal.

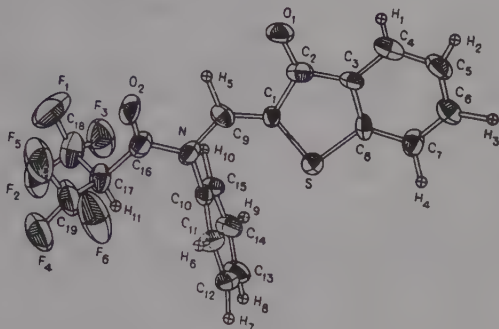


Fig. 1. The  $C_{19}H_{11}F_6NO_2S$  molecule.

2,5-DIPHENYL-3,4-DIMETHYLENE-1,6,6a-TRITHIAPENTALENE  
 $C_{19}H_{14}S_3$

B. BIRKNES, A. HORDVIK and L.J. SAETHRE, 1982. Acta Chem. Scand., A36, 683-688.

Orthorhombic,  $Pnma$ ,  $a = 7.165$ ,  $b = 29.704$ ,  $c = 7.372$  Å,  $D_m = 1.427$ ,  $Z = 4$ . Mo radiation,  $R = 0.037$  for 934 reflexions.

The molecule has crystallographic  $m$  symmetry, atoms  $C(3a)$  and  $S(6a)$  lying on the mirror plane (Fig. 1). The central ring system is almost planar and the phenyl rings are twisted  $30^\circ$  about their respective connecting bonds. The  $S-S$  bond distances are  $2.351$  Å and the  $S(1)-S(6a)-S(6)$  angle is  $173.43^\circ$ . Other bond lengths are:  $S(1)-C(2)$   $1.725$ ,  $S(6a)-C(3a)$   $1.716$ ,  $C(2)-C(3)$   $1.374$ ,  $C(3)-C(3a)$   $1.407$ ,  $C(3)-C(7)$   $1.529$  and  $C(7)-C(8)$   $1.566$  Å. The bonds connecting the phenyl rings to the central ring system are  $1.489$  Å in length. These lengths have been corrected for libration.

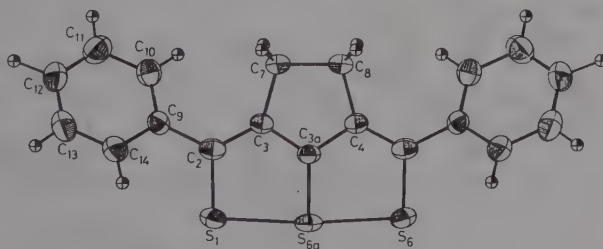


Fig. 1. Molecular structure of  $C_{19}H_{14}S_3$ .

THIANTHRENIUM BISCARBETHOXYMETHYLIDE  
 $C_{19}H_{18}O_4S_2$

A.L. TERNAY, Jr., J.C. BAACK, S.S.C. CHU, V. NAPOLEONE, G. MARTIN and C. ALFARO, 1982. J. Heterocyclic Chem., 19, 833-836.

Triclinic,  $P\bar{1}$ ,  $a = 8.092$ ,  $b = 10.389$ ,  $c = 11.374$  Å,  $\alpha = 95.55$ ,  $\beta = 108.17$ ,  $\gamma = 104.09^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.071$  for 2179 reflexions.

The angle of fold between aryl rings (Fig. 1) is  $135.7^\circ$ , the nearly-planar malonylide fragment bisecting this angle. The carbonyl oxygen of the endo carboxy group is anti to the nonbonding electron pair on sulfur.

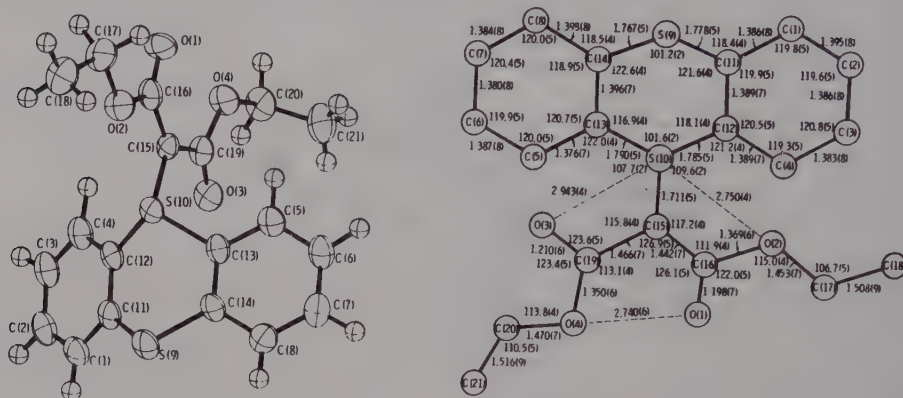


Fig. 1. The  $C_{19}H_{16}O_4S_2$  molecule and dimensions.

(Z)-4-METHYL-6-PHENYL-2-(THIOBENZOYLMETHYLENE)-2H-THIAPYRAN  
 $C_{20}H_{16}S_2$

J. KAISER, A. HANTSCHMANN, R. RICHTER, R. SCHEIBE and J. FABIAN, 1982. *Tetrahedron*, **38**, 1639-1645.

Monoclinic,  $P2_1/c$ ,  $a = 7.813$ ,  $b = 18.381$ ,  $c = 11.302$  Å,  $\beta = 90.48^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.04$  for 3427 reflexions.

The molecule is shown in Fig. 1. The no-bond interaction between S atoms ( $S \cdots S$  3.000(1) Å) is caused by  $\pi$ -type rather than by Coulomb-type interaction.

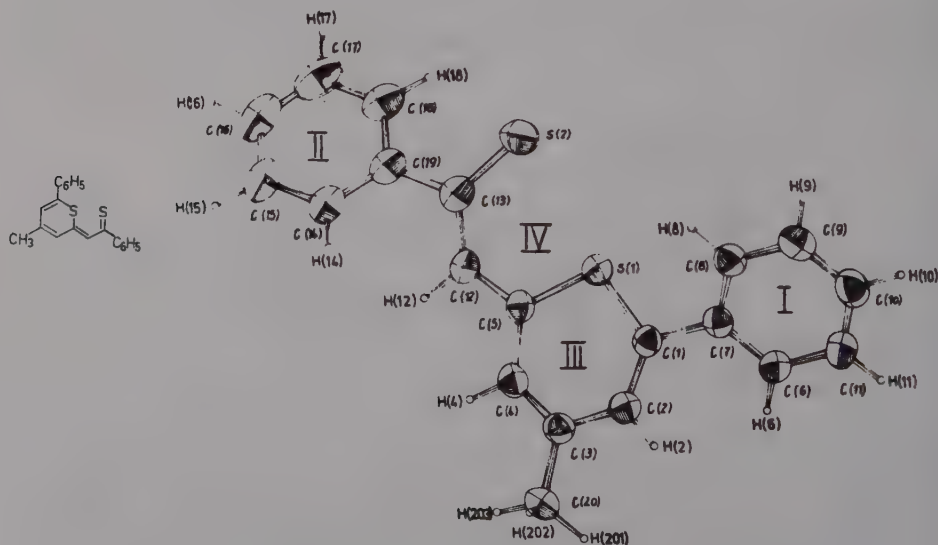


Fig. 1. The  $C_{20}H_{16}S_2$  molecule.

9-HYDROXY-1,4-DIMETHYLTHIOXANTHENIUM BIS(CARBOMETHOXY)METHYLIDE  
 $C_{20}H_{20}O_5S$  (I)

1,4-OXATHIANIUM BIS(CARBOMETHOXY)METHYLIDE  
 $C_9H_{14}O_5S$  (II)

M.A. ABBADY, S. ASKARI, M. MORGAN, A.L. TERNAY, Jr., J. GALLOY and W.H. WATSON, 1982. *J. Heterocyclic Chem.*, **19**, 1473-1479.

I. Triclinic,  $P1$ ,  $a = 8.384$ ,  $b = 8.653$ ,  $c = 16.378$  Å,  $\alpha = 119.10$ ,  $\beta = 92.53$ ,  $\gamma = 115.64^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.036$  for three-dimensional data.

II. Orthorhombic,  $Pnma$ ,  $a = 19.312$ ,  $b = 6.917$ ,  $c = 8.421$  Å,  $Z = 4$ . Cu radiation,  $R = 0.044$  for three-dimensional data.

The two phenyl rings in I (Fig. 1) have an interplanar angle of  $158.7(2)^\circ$ . The six-membered ring containing the sulfur atom is in a boat conformation with the C(9) and S(10) substituents occupying axial sites. The carbanion moiety is planar and the axial conformation about the S(10)-C(15) bond is determined by electrostatic repulsion between the sulfur lone pair and the  $\pi$ -system of the carbanion and is stabilized by an intramolecular hydrogen bond  $O(9)-H...O(21)$   $2.725(2)$  Å. In II (Fig. 1) a crystallographic mirror plane bisects the 1,4-oxathiane ring while the carbanion moiety lies entirely within the plane. The six-membered ring exhibits a chair conformation with the carbanion occupying an equatorial site. The conformation about the S(10)-C(15) bond is again determined by electrostatic repulsion between the sulfur lone pair and the  $\pi$ -system of the carbanion. The S-C(15) distance is  $1.718(3)$  Å in I and  $1.723(2)$  Å in II.

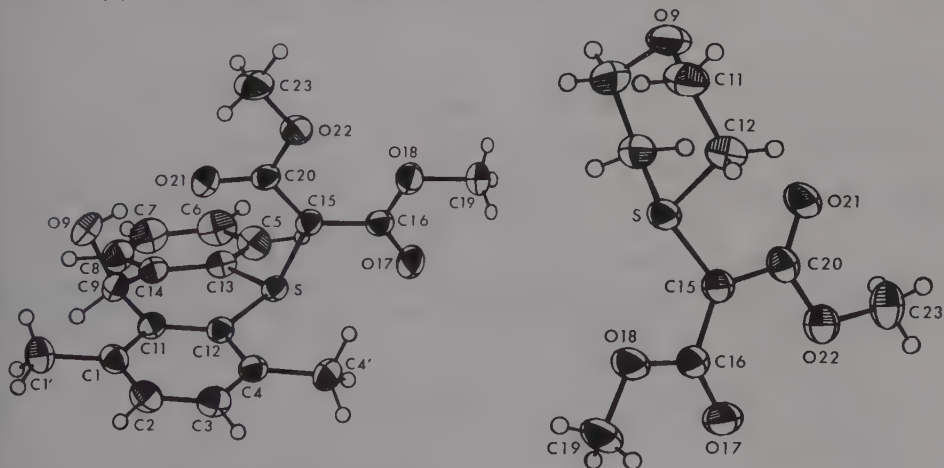


Fig. 1. Views of the bis(carbomethoxy)methylide derivatives I, left; II, right.

TETRAMETHYLTETRATHIAFULVALENE HEXAFLUOROARSENATE  
 $C_{20}H_{24}AsF_6S_8$

B. LIAUTARD, S. PEYTAVIN, G. BRUN and M. MAURIN, 1982. *Cryst. Struct. Comm.*, **11**, 1841-1843.

Triclinic,  $P\bar{1}$ ,  $a = 7.178$ ,  $b = 7.610$ ,  $c = 13.317$  Å,  $\alpha = 82.03$ ,  $\beta = 95.75$ ,  $\gamma = 107.11^\circ$ ,  $Z = 1$ . Mo radiation,  $R = 0.041$  for 1341 reflexions.

The TMTF units (Fig. 1) are nearly perpendicular to  $a$  and repeat by inversion, so that the consecutive distances between their planes ( $3.54$  and  $3.64$  Å) are crystallographically independent. The closest S...S interstack distance is  $4.01$  Å.



The hexafluoroarsenate anions are distributed along lines parallel to *a*, with a translation of 7.178 Å. The closest S...F distance is 3.27 Å.

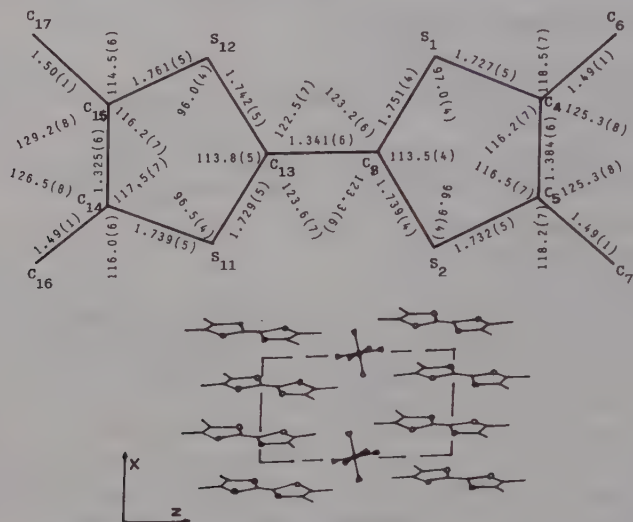
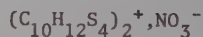
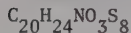


Fig. 1. Cation dimensions and a view of the packing in  $C_{20}H_{24}AsF_6S_8$ .

#### TETRAMETHYLTETRATHIAFULVALENE NITRATE



B. LIAUTARD, S. PEYTAVIN, G. BRUN and M. MAURIN, 1982. Acta Cryst., B38, 2746-2749.

Triclinic,  $P\bar{1}$ ,  $a = 7.061$ ,  $b = 7.395$ ,  $c = 12.547$  Å,  $\alpha = 89.50^\circ$ ,  $\beta = 93.91^\circ$ ,  $\gamma = 108.97^\circ$ ,  $D_m = 1.553$ ,  $Z = 1$ . Mo radiation,  $R = 0.042$  for 1588 reflexions.

In the crystal structure (Fig. 1) the tetramethyltetrathiafulvalene units are nearly perpendicular to the *a* axis; they are not crystallographically equivalent and form a diadic system. The nitrate ion is disordered and lies on a centre of symmetry.

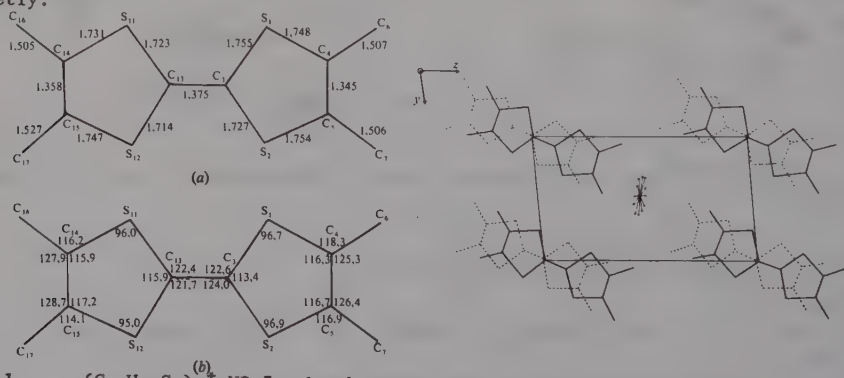


Fig. 1.  $(C_{10}H_{12}S_4)_2^+, NO_3^-$ : bond lengths and angles in the tetramethyltetrathiafulvalene cation and a view of the crystal structure down the *a* axis.

DI(4,4',5,5'-TETRAMETHYL- $\Delta^2,2'$ -DI-1,3-DISELENOYLIDEN)IUM NITRATE  
 $C_{20}H_{24}NO_3Se_8$

H. SOLING, G. RINDORF and N. THORUP, 1982. Cryst. Struct. Comm., 11, 1975-1980.

Triclinic,  $P\bar{1}$ ,  $a = 7.217$ ,  $b = 7.567$ ,  $c = 12.822$  Å,  $\alpha = 89.14$ ,  $\beta = 86.61$ ,  $\gamma = 70.56^\circ$ ,  $D_m = 2.40$ ,  $Z = 1$ . Mo radiation,  $R = 0.043$  for 720 reflexions.

Two independent cations lie on inversion centres and have approximate  $mmm$  symmetry. The  $NO_3^-$  ion is disordered about an inversion centre. In the cation stacks (Fig. 1) the interplanar distances are 3.60 and 3.61 Å. The Se-C bond lengths are in the range 1.862(13) to 1.907(13) Å.

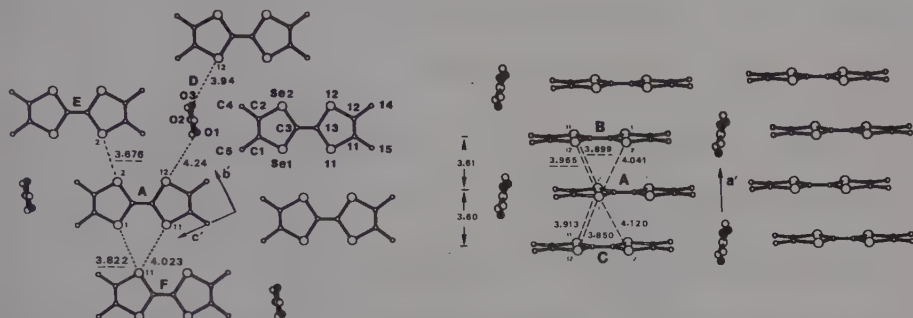


Fig. 1. Two views of the packing of  $C_{20}H_{24}NO_3Se_8$ .

DI(4,4',5,5'-TETRAMETHYL- $\Delta^2,2'$ -BI-1,3-DISELENOYLIDEN)IUM PERRHENATE

$C_{20}H_{24}O_4ReSe_8$  (I)  $2C_{10}H_{12}Se_4^{0.5+}, ReO_4^-$

DI(4,4',5,5'-TETRAMETHYL- $\Delta^2,2'$ -BI-1,3-DISELENOYLIDEN)IUM PERCHLORATE

$C_{20}H_{24}ClO_4Se_8$  (II)  $2C_{10}H_{12}Se_4^{0.5+}, ClO_4^-$

G. RINDORF, H. SOLING and N. THORUP, 1982. Acta Cryst., B38, 2805-2808.

I. Triclinic,  $P\bar{1}$ ,  $a = 7.284$ ,  $b = 7.751$ ,  $c = 13.483$  Å,  $\alpha = 83.23$ ,  $\beta = 86.56$ ,  $\gamma = 70.08^\circ$ ,  $D_m = 2.77$ ,  $Z = 1$ . Mo radiation,  $R = 0.045$  for 1883 reflexions (at 295 K);  $a = 7.154$ ,  $b = 7.710$ ,  $c = 13.310$  Å,  $\alpha = 83.88$ ,  $\beta = 88.41$ ,  $\gamma = 69.12^\circ$ . Mo radiation,  $R = 0.044$  for 694 reflexions (at 120 K).

II. Triclinic,  $P\bar{1}$ ,  $a = 7.266$ ,  $b = 7.678$ ,  $c = 13.275$  Å,  $\alpha = 84.58$ ,  $\beta = 86.73$ ,  $\gamma = 70.43^\circ$ ,  $D_m = 2.39$ ,  $Z = 1$ . Mo radiation,  $R = 0.055$  for 2285 reflexions (at 295 K).

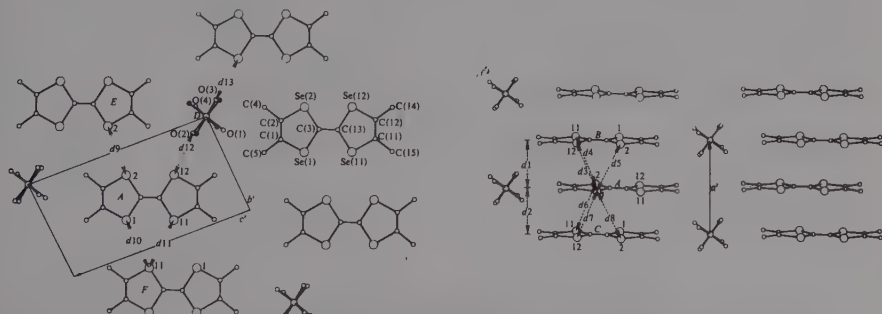


Fig. 1.  $C_{20}H_{24}O_4ReSe_8$  (I) and  $C_{20}H_{24}ClO_4Se_8$  (II): views of the crystal structure along  $a$  and of the stacking of the molecules in the crystal.

Both compounds (Fig. 1) belong to a series of mixed-valence cation-radical salts and are isostructural with the corresponding hexafluorophosphate (1) which has similar bond lengths and angles. The  $\text{ReO}_4^-$  and  $\text{ClO}_4^-$  anions have a distorted tetrahedral shape and are disordered in the crystal; at 120 K there appears to be an ordering of the  $\text{ReO}_4^-$  anions. The cations are stacked in the crystal in zigzag fashion along the short a axis with the stacks arranged in sheets parallel to the ab plane.

1. Structure Reports, 48B, 426.

3-METHOXY-10-METHYL-11-PHENYLDIBENZO[b,f]THIEPINE

$\text{C}_{22}\text{H}_{18}\text{OS}$  (I)

3-ALLYLOXY-10-ETHYL-11-PHENYLDIBENZO[b,f]OXEPINE

$\text{C}_{25}\text{H}_{22}\text{O}_2$  (II)

G. BANDOLI and M. NICOLINI, 1982. J. Cryst. Spectrosc. Res., 12, 425-447.

I. Tetragonal,  $I4_1/a$ ,  $a = 33.81$ ,  $c = 6.065 \text{ \AA}$ ,  $D_m = 1.26$ ,  $Z = 16$ . Mo radiation,  $R = 0.090$  for 784 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 12.115$ ,  $b = 16.316$ ,  $c = 10.136 \text{ \AA}$ ,  $\beta = 105.05^\circ$ ,  $D_m = 1.23$ ,  $Z = 4$ . Mo radiation,  $R = 0.083$  for 442 reflexions.

The dihedral angle (Fig. 1) between the phenyl-ring mean planes is  $111.3^\circ$  in (I) and  $121.1^\circ$  in (II), the middle seven-membered ring has the "boat" conformation, and the tricyclic moiety has twist and skew values of  $6^\circ$  and  $0.42 \text{ \AA}$  in (I) and  $0.3^\circ$  and  $0.79 \text{ \AA}$  in (II). The overall conformational characteristic for the tricyclic (6,7,6)-dibenzo[b,f]heteroepin derivatives are reviewed to gain understanding of what requirements may be important for interaction of drugs of this class at the receptor site.

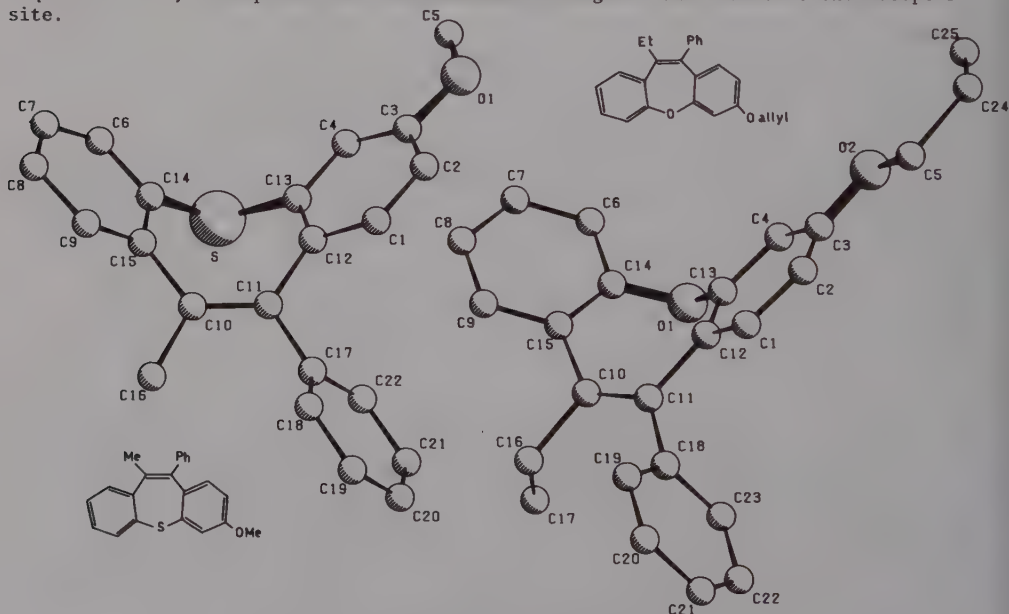
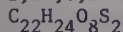


Fig. 1. Views of the dibenzo[b,f]heteroepin compounds.

## 2,2',6,6'-TETRAETHOXYCARBONYL-4,4'-BITHIAPYRANYLIDENE



B.F. DAROCHA, D.D. TITUS, D.J. SANDMAN and D.E. WARNER, 1982. *Acta Cryst.*, **B38**, 2267-2269.

Orthorhombic, *Ib*am,  $a = 23.762$ ,  $b = 14.486$ ,  $c = 6.788$  Å,  $D_m = 1.36$ ,  $Z = 4$ . Mo radiation,  $R = 0.057$  for 609 reflexions.

The molecule (Fig. 1) has  $2/m$  crystallographic symmetry which requires planarity or disorder in the lattice; the planar model provides an adequate fit to the data. The central "double" bond, C(5)-C(5') is long, 1.405(8) Å. The molecular planes lie perpendicular to the  $c$  axis, the interplanar spacing being  $c/2$  (3.394 Å). The S-S axes of neighbouring molecules are staggered with respect to each other by 88°.

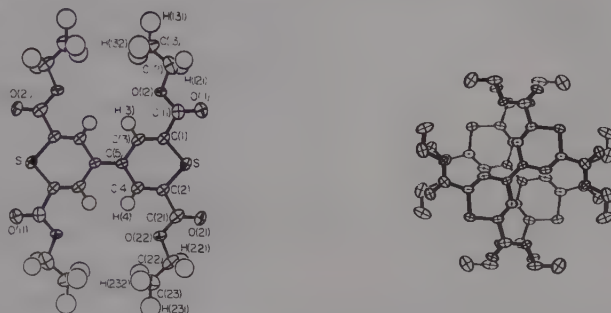
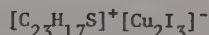
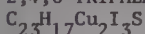


Fig. 1.  $\text{C}_{22}\text{H}_{24}\text{O}_8\text{S}_2$ : a perspective view of the molecule and a projection looking along the  $c$  axis.

## 2,4,6-TRIPHENYLTHIOPYRILIUM DICOPPER TRIIODIDE



A.S. BATANOV, Yu.T. STRUCHKOV, L.Yu. UKHIN and N.A. DOLGOPOLOVA, 1982. *Inorg. Chim. Acta*, **63**, 17-22.

Hexagonal,  $P6_1$ ,  $a = 13.856$ ,  $c = 20.778$  Å,  $D_m = 2.29$ ,  $Z = 6$ . Mo radiation,  $R = 0.033$  for 2872 reflexions (at  $-120^\circ\text{C}$ ).

The structure contains infinite anion chains  $n[\text{Cu}_2\text{I}_3]^-$  and discrete triphenylthiopyrilium cations (Fig. 1). The chains of  $\text{I}_4$  tetrahedra are centred by Cu atoms and joined by their faces and edges. The shortest Cu...Cu distances are 2.479 and 2.634 Å. The cation is almost parallel to the (001) plane and is disordered over three orientations so that the S atom occupies positions 1, 3 and 5 with unequal occupancy factors. As a result the accuracy of the heterocycle geometrical parameters is low. The planes of the phenyl substituents in the 2, 4 and 6 positions form dihedral angles of 16.6, 15.8 and 15.9° respectively with the mean plane of the heterocycle.

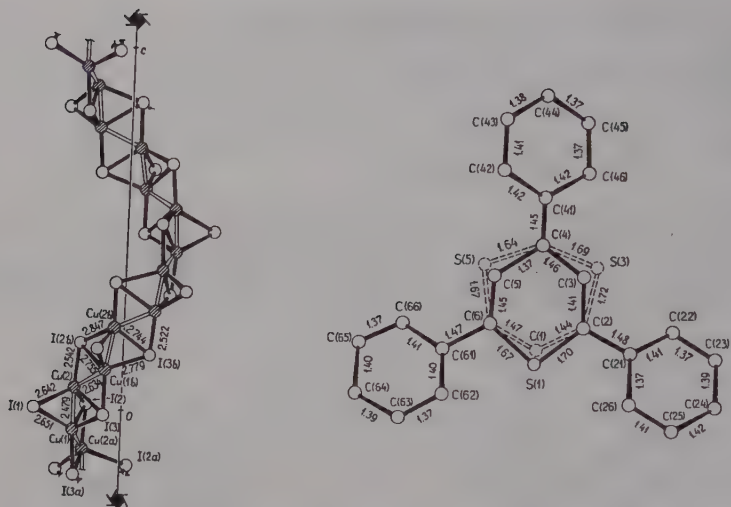


Fig. 1. Projection of anionic chain in  $C_{23}H_{17}Cu_2I_3S$  on 010 plane (left) and projection of cation on (001) plane (right).

anti-DITHIA[3.3](2,6)TRIQUINACENOPHANE  
 $C_{24}H_{24}S_2$

G. SHOHAM, W.P. ROBERTS and W.N. LIPSCOMB, 1982. Cryst. Struct. Comm., 11, 2075-2082.

Triclinic,  $P\bar{1}$ ,  $a = 7.679$ ,  $b = 7.766$ ,  $c = 9.730$  Å,  $\alpha = 93.58$ ,  $\beta = 111.91$ ,  $\gamma = 111.62^\circ$ ,  $Z = 1$ . Mo radiation,  $R = 0.044$  for 2497 reflexions.

The molecule (Fig. 1) lies about an inversion centre and is the anti-isomer. The two triquinacene units are concave inwards and in a relatively staggered conformation.

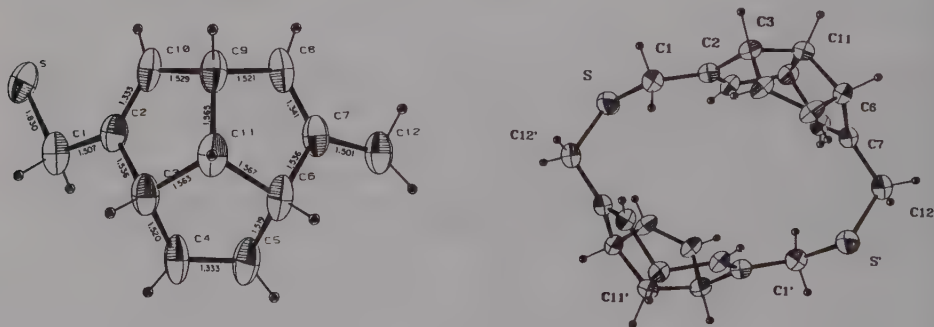


Fig. 1. Bond lengths in the asymmetric unit of  $C_{24}H_{24}S_2$  and a view of the centrosymmetric molecule.

DIMETHYL 6,8-DIPHENYL-6H-CYCLOHEPTA[b]THIOPHENE-4,5-DICARBOXYLATE  
 $C_{25}H_{20}O_4S$

J. KRAMSTAD, T. SLETTEN and S. NORDENSON, 1982. Chem. Scr., 20, 74-77.

Monoclinic,  $P2_1/c$ ,  $a = 6.911$ ,  $b = 12.497$ ,  $c = 23.902$  Å,  $\beta = 92.93^\circ$ ,  $D_m = 1.1$ ,  $Z = 4$ . Mo radiation,  $R = 0.070$  for 3995 reflexions (at 130 K).

The thiophene ring and the double-bonded carbon atoms (C(4), C(5), C(7) and C(8)) constitute a planar arrangement whereas the atom C(6) lies above (or below this plane) (Fig. 1). The position of the 6-phenyl group is unexpected; it occupies a pseudo-axial conformation and is bent inwards under (or above) the thiophene ring. Some distances of interest are: S-C(2) 1.709, S-C(8a) 1.724, C(2)-C(3) 1.354, C(3)-C(3a) 1.430, C(3a)-C(8a) 1.396, C(3a)-C(4) 1.470, C(4)-C(5) 1.362, C(5)-C(6) 1.540, C(6)-C(7) 1.509, C(6)-C(15) 1.533, C(7)-C(8) 1.343, C(8)-C(8a) 1.462 and C(8)-C(9) 1.499 Å.

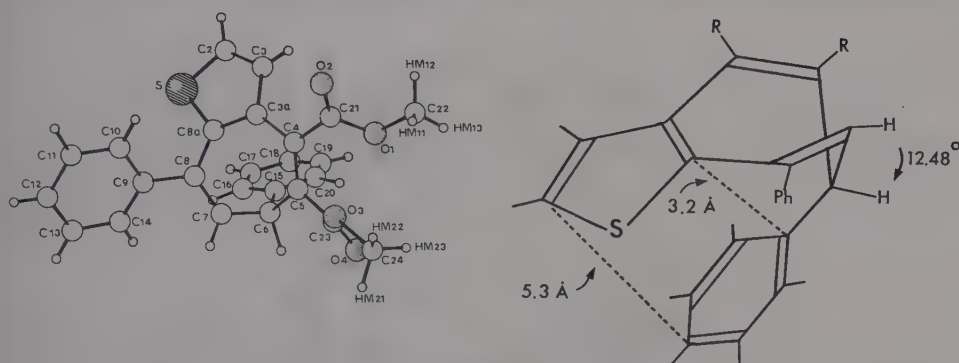


Fig. 1. Molecular structure of  $C_{25}H_{20}O_4S$  (left), and its preferred conformation (right).

SPIRO[9H-FLUORENE-9,3'(10'6H)]-FLUORENO[9,1-cd][1,2]DITHIIN  
 $C_{26}H_{16}S_2$

S. SCHEIBYE, R. SHABANA, S.-O. LAWESSON and C. ROMMING, 1982. *Tetrahedron*, **38**, 993-1001.

Monoclinic,  $P2_1/n$ ,  $a = 11.989$ ,  $b = 11.564$ ,  $c = 14.038$  Å,  $\beta = 100.77^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.036$ .

A view of the structure is in Fig. 1. The sulphur atoms S1 and S2 are respectively 0.18 Å below and 0.98 Å above the plane through C13, C24, C25, C26. The torsion angle C-S-S-C is  $63.2^\circ$ .

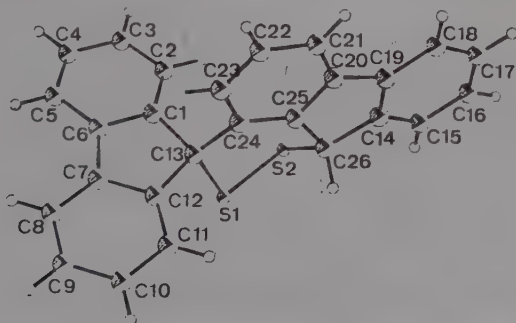


Fig. 1. A view of  $C_{26}H_{16}S_2$ .



## CYCLOPENTA(p-PHENYLENE SULPHIDE)



M.L. KAPLAN, W.D. REENTS Jr. and C.S. DAY, 1982. *Cryst. Struct. Comm.*, **11**, 1751-1758.

Monoclinic,  $\text{C2/c}$ ,  $a = 36.796$ ,  $b = 13.173$ ,  $c = 11.400$  Å,  $\beta = 108.70^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.042$  for 3187 reflexions.

In this cyclic molecule (Fig. 1), no two benzene rings are coplanar and no four sulphur atoms are coplanar. The average C-S bond length is  $1.778(4)$  Å. The C-S-C angles vary between  $100.1(2)^\circ$  and  $104.0(2)^\circ$  with an average value  $102.5(2)^\circ$ .

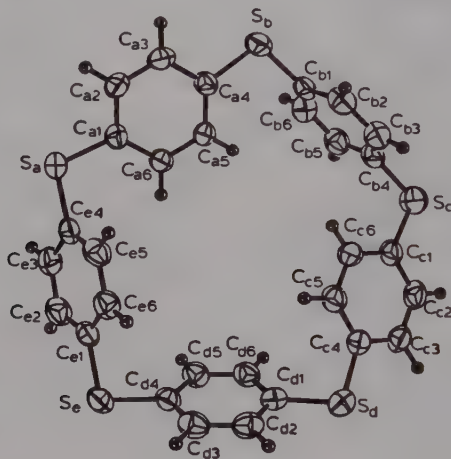
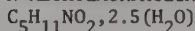


Fig. 1. The  $\text{C}_{30}\text{H}_{20}\text{S}_5$  molecule.

## N-METHYLMORPHOLINE N-OXIDE HYDRATE



E. MAIA and S. PÉREZ, 1982. *Acta Cryst.*, **B38**, 849-852.

Monoclinic,  $\text{P2}_1/\text{c}$ ,  $a = 12.803$ ,  $b = 6.500$ ,  $c = 21.913$  Å,  $\beta = 109.99^\circ$ ,  $D_m = 1.22$ ,  $Z = 8$ . Cu radiation,  $R = 0.042$  for 991 reflexions.

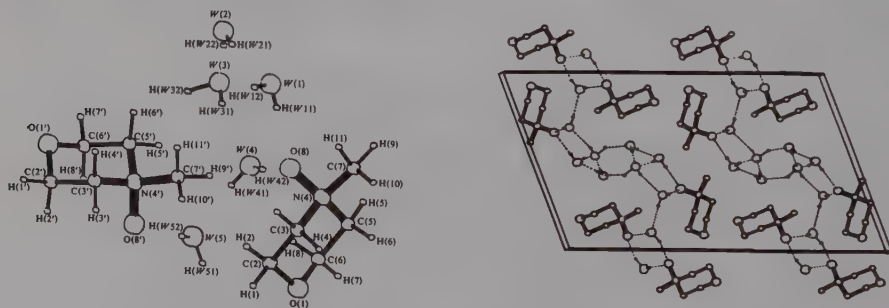


Fig. 1.  $\text{C}_5\text{H}_{11}\text{NO}_{4.5}$ : views of the asymmetric unit (left) and of the packing of the molecules in the crystal (right); hydrogen bonds are shown as dashed lines.

The structure (Fig. 1) contains an intricate network of hydrogen bonding involving ten non-equivalent  $\text{O} \cdots \text{O}$  distances in the range  $2.661$ – $2.810$  Å. The

morpholine rings take the chair form with axial N-O (1.392 and 1.399(3) Å). The non-solvent character of the compound is attributed to the saturation of the N-oxide by hydrogen bonds.

4-AMINO-5-NITROBENZO[1,2-c:3,4-c']BIS[1,2,5]OXADIAZOLE 3,8-DIOXIDE  
 $C_6H_2N_6O_6$

H.L. AMMON and S.K. BHATTACHARJEE, 1982. Acta Cryst., B38, 2498-2502.

Orthorhombic,  $P2_12_12_1$ ,  $a = 13.6644$ ,  $b = 6.6969$ ,  $c = 9.5462$  Å,  $Z = 4$ . Cu radiation,  $R = 0.034$  for 747 reflexions.

The molecule (Fig. 1) is approximately planar with the largest out-of-plane deviations associated with the  $NH_2$ ,  $NO_2$  and two oxide groups. Bond lengths and angles are normal. Benzene annelation has no significant effect on the basic oxadiazole structure. The molecules pack in sheets approximately perpendicular to  $c$ , the shortest contact being a 2.940(5) Å C...O. Hydrogen bonding by one of the amino hydrogen atoms is limited to a strong intramolecular N-H...O contact with the neighbouring nitro group, while the other hydrogen enters into somewhat weaker intra- and intermolecular contacts with oxygen.

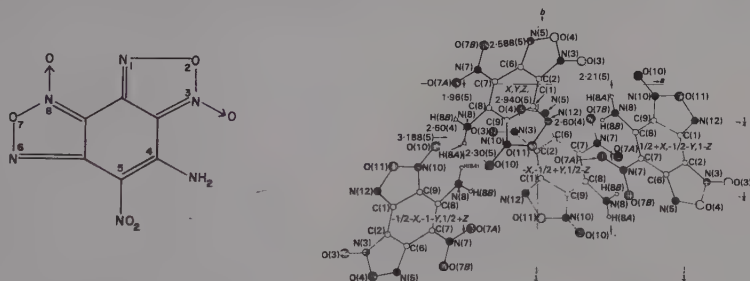


Fig. 1.  $C_6H_2N_6O_6$ : structural formula of the molecule and molecular packing in the crystal, as viewed down  $c$ ; some contact distances are given.

TETRAMETHYLENEFURAZAN N-OXIDE



M.J. BARROW, 1982. Acta Cryst., B38, 308-310.

Orthorhombic,  $Pbca$ ,  $a = 9.54$ ,  $b = 12.61$ ,  $c = 10.69$  Å (at 125K),  $Z = 8$ . Cu radiation,  $R = 0.042$  for 800 reflexions (photographic data).

In the molecule (Fig. 1) the furazan N-oxide ring is disordered and this prevents decisive comparison of its geometry with that found in trimethylenefurazan N-oxide (1). Nonetheless it appears that addition of a further methylene group alleviates the angle strain in the carbocycle and the O(1)-N(2) bond shortens from 1.494 Å (1) to 1.465 Å (title compound).

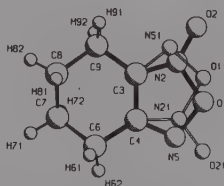


Fig. 1. View of the  $C_6H_8N_2O_2$  molecule showing disordering of the furazan N-oxide ring.

1. Structure Reports, 45B, 394.

N-(2-AMONIOETHYL)MORPHOLINIUM TETRACHLOROCUPRATE(II)

 $C_6H_{16}Cl_4CuN_2O$  $(C_6H_{16}N_2O)^{2+}(CuCl_4)^{2-}$ L.P. BATTAGLIA, A. BONAMARTINI CORRADI, G. MARCOTRIGIANO, L. MENABUE and G.C. PELLACANI, 1982. *Inorg. Chem.*, 21, 3919-3922.

green form:

Monoclinic,  $I2/a$ ,  $a = 15.934$ ,  $b = 13.023$ ,  $c = 14.069$  Å,  $\beta = 118.89^\circ$ ,  $D_m = 1.74$ ,  $Z = 8$ . Mo radiation,  $R = 0.047$  for 1453 reflexions.

yellow form:

Monoclinic,  $P2_1/a$ ,  $a = 15.909$ ,  $b = 12.420$ ,  $c = 6.355$  Å,  $\beta = 98.77^\circ$ ,  $D_m = 1.78$ ,  $Z = 4$ . Mo radiation,  $R = 0.0445$  for 2165 reflexions.

The structures of the two forms are shown in Fig. 1. In the green crystals two crystallographically independent  $[CuCl_4]^{2-}$  anions are present, one with a flattened tetrahedral geometry (Cu-Cl 2.240(1) and 2.314(1) Å) and the other square planar (Cu-Cl 2.269(1) and 2.266(3) Å). In the yellow form only distorted tetrahedral  $[CuCl_4]^{2-}$  anions are present (Cu-Cl 2.241(1)-2.259(1) Å). In both forms, the anions are H-bonded to cations having normal dimensions, the H-bonding being stronger in the green modification.

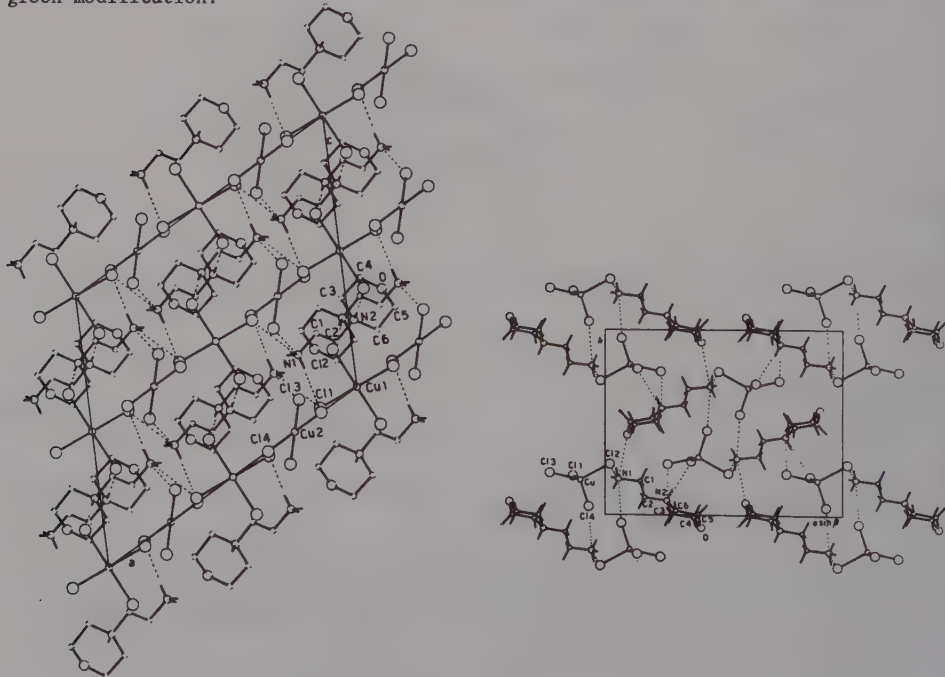
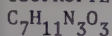


Fig. 1. Structures of the green (left) and yellow (right) forms of  $(C_6H_{16}N_2O)^{2+}(CuCl_4)^{2-}$ .

## ISOPROPYL 4-METHYL-3-FURAZANCARBAMATE



M. CALLERI, G. CHIARI, A.G. MANFREDOTTI and D. VITERBO, 1982. *Acta Cryst.*, **B38**, 3116-3118.

Monoclinic,  $P2_1/c$ ,  $a = 9.14$ ,  $b = 11.91$ ,  $c = 8.92 \text{ \AA}$ ,  $\beta = 91.3^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.048$  for 1543 reflexions.

The furazan and carbamate moieties (Fig. 1) are planar, the two mean planes making an angle of  $7.3(2)^\circ$ . The former is slightly asymmetric because of a difference between the two N-O bonds. A comparison is given of the dimensions of the furazan ring in disubstituted derivatives.

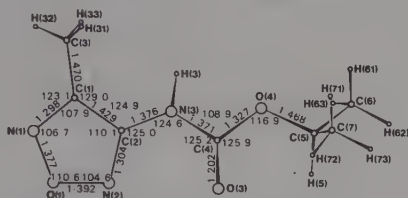
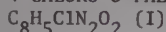
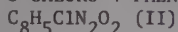


Fig. 1. Dimensions for  $\text{C}_7\text{H}_{11}\text{N}_3\text{O}_3$  ( $\sigma$  0.002-0.004  $\text{\AA}$ , 0.3-0.4 $^\circ$ ).

## 4-CHLORO-3-PHENYLFUROXAN



## 3-CHLORO-4-PHENYLFUROXAN



D. VITERBO, G. CHIARI and R. CALVINO, 1982. *Acta Cryst.*, **B38**, 3045-3049.

I. Orthorhombic,  $Pbca$ ,  $a = 17.156$ ,  $b = 12.803$ ,  $c = 7.835 \text{ \AA}$ ,  $Z = 8$ . Cu radiation,  $R = 0.064$  for 985 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 14.011$ ,  $b = 9.713$ ,  $c = 14.198 \text{ \AA}$ ,  $\beta = 119.66^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.048$  for 1949 reflexions.

In isomer II (Fig. 1) the asymmetric unit is formed by two independent molecules, which show some remarkable differences in the geometry of the furoxan ring; dipole-dipole interactions between the almost-facing bonds C-Cl and N $\rightarrow$ O and the different planarity of the chlorofuroxan groups can account for these differences, while the former effect can explain the tendency of this isomer to undergo isomerization to form A. The phenyl group is rotated with respect to the five-membered ring by  $27.7(1)^\circ$  and  $25.4(1)^\circ$  in the two molecules; in isomer I this angle increases to  $44.1(1)^\circ$ .

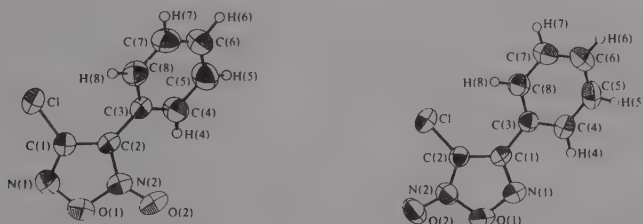


Fig. 1. The isomers (I) and (II) of  $\text{C}_8\text{H}_5\text{ClN}_2\text{O}_2$ .

(2S,4S,6R)-2,6,7,9-TETRAHYDRO-2,4,6-TRIS(TRICHLOROMETHYL)-8H-[1,3,5] TRIAZINO[1,2-c]-[1,3,5]OXADIAZIN-8-ONE DIOXANE SOLVATE  
 $C_8H_5Cl_9N_4O_2 \cdot 2(C_4H_8O_2)$

K.H. PILGRAM, R.D. SKILES, E.J. SILVEIRA, L.H. GALE and G.E. POLLARD, 1982. J. Org. Chem., 47, 3046-3049.

Orthorhombic, Pccn,  $a = 18.94$ ,  $b = 30.798$ ,  $c = 11.550$  Å,  $Z = 8$ . Cu radiation,  $R = 0.12$  for 4413 reflexions.

In the molecule (Fig. 1) there are two major planar regions: namely, the atoms of the s-triazine moiety (with the exception of carbon 4) and the atoms of the 1,3,5-oxadiazine portion (with the exception of carbon 6). The dihedral angle between the planes of the two rings is  $10.4^\circ$ . The trichloromethyl groups exist in an all-axial alternating trans arrangement. The bond distance between the bridgehead carbon atom (C-6) and the nitrogen atom (N-3) of the oxadiazine ring portion is considerably shorter (1.29 Å) than that between C-6 and the nitrogen atom (N-2) of the s-triazine ring moiety (1.37 Å), indicating a higher degree of double bond character between C-6 and N-3 than between C-6 and N-2.

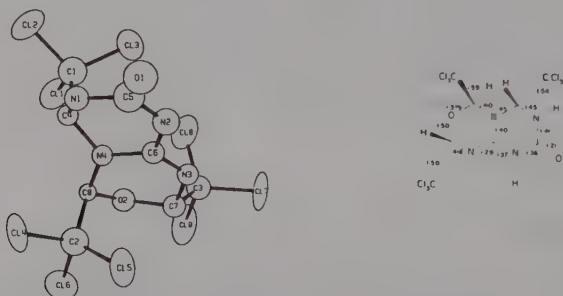


Fig. 1. The oxadiazin-8-one and bond lengths.

5-CHLORO-3-METHYL-1,2-BENZISOXAZOLE 2-OXIDE  
 $C_8H_6ClNO_2$

G. CHIARI and D. VITERBO, 1982. Acta Cryst., B38, 323-325.

Monoclinic,  $P2_1/n$ ,  $a = 16.329$ ,  $b = 5.668$ ,  $c = 16.957$  Å,  $\beta = 91.87^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.033$  for 1365 reflexions.

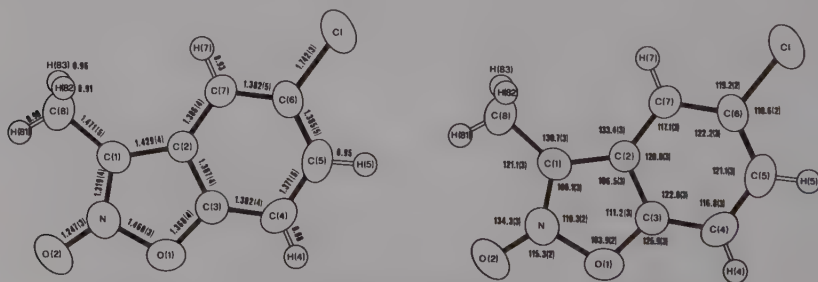


Fig. 1.  $C_8H_6ClNO_2$ : average bond distances and angles taken over the two independent molecules.

The crystal structure contains two crystallographically independent molecules (Fig. 1) which are almost parallel to each other and have similar molecular geometry, with planar benzisoxazole fragments. The compound resembles furoxans in two ways: first, the N-O(1) distance (1.468 Å) is lengthened and is comparable with the most strained bonds (1.425-1.501 Å) found in some fused furoxans, and secondly, the

N-O(2) distance (1.247 Å) falls only just outside the range (1.204-1.242 Å) found in furoxans. Intermolecular contacts correspond to van der Waals interactions.

### 2,4-DIHYDROXY-2H-1,4-BENZOXAZIN-3-ONE



L. KUTSCHABSKY, W. DATHE and G. SCHNEIDER, 1982. Cryst. Struct. Comm., 11, 841-846.

Triclinic,  $P\bar{1}$ ,  $a = 10.415$ ,  $b = 10.512$ ,  $c = 7.352$  Å,  $\alpha = 90.19$ ,  $\beta = 99.03$ ,  $\gamma = 90.91^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.086$  for 2482 reflexions.

Bond lengths and torsion angles for the two independent molecules (Fig. 1) are similar and close to normal values. The molecules are linked by O-H...O hydrogen bonds (2.603-2.791 Å).

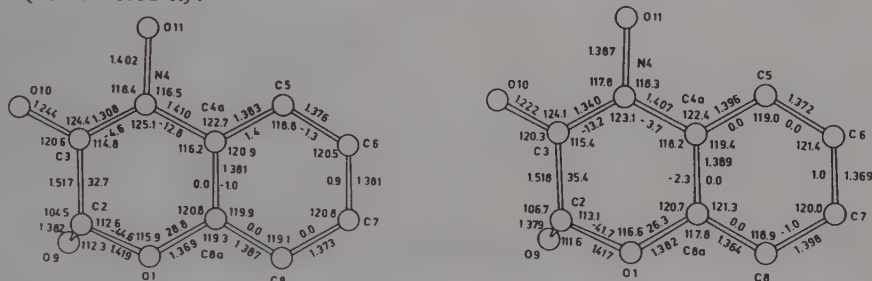
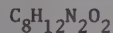


Fig. 1. Bond distances ( $\sigma$  0.003-0.007 Å) and some torsion angles for the  $\text{C}_8\text{H}_7\text{NO}_4$  molecule.

### HEXAMETHYLENEFURAZAN N-OXIDE



M.M. HARDING and R.M. PATON, 1982. Acta Cryst., B38, 1395-1397.

Monoclinic,  $P2_1/n$ ,  $a = 11.50$ ,  $b = 10.50$ ,  $c = 7.07$  Å,  $\beta = 101.0^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.056$  for 523 reflexions (photographic data).

In the molecule (Fig. 1) the C(3)-N(2)-O(2) angle of  $136.2^\circ$  is very large, as is also found in the tetra- and trimethylene analogues (1,2). The N(2)-O(1) bond distance is 1.428 Å. Atoms O(1), N(2), C(3), C(4) and N(5) are coplanar (within 0.005 Å), but O(2) is 0.028 Å from their plane.

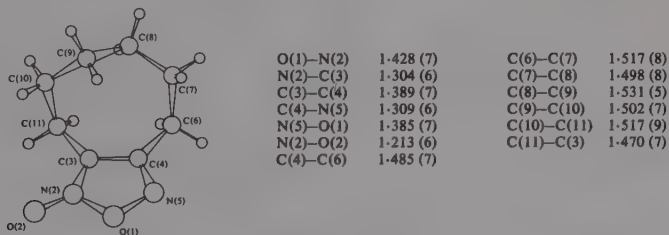


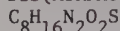
Fig. 1.  $\text{C}_8\text{H}_{12}\text{N}_2\text{O}_2$ : a perspective view of the molecule and bond lengths.

1. Structure Reports, this volume, p423.

2. Ibid., 45B, 394.



## BIS(MORPHOLINO)SULPHIDE



C. RØMMING, G.O. NEVSTAD and J. SONGSTAD, 1982. Acta Chem. Scand., A36, 399-405.

Monoclinic,  $P2_1/c$ ,  $a = 11.207$ ,  $b = 15.658$ ,  $c = 11.574$  Å,  $\beta = 90.19^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.050$  for 2373 reflexions (at  $-150^\circ\text{C}$ ).

The geometry of the two molecules in the asymmetric unit is nearly identical. The molecule possesses a pseudo twofold axis, with the morpholino rings in the chair conformation and linked equatorially to the central S atom (Fig. 1). The  $\text{N}(1)\text{-S-N}(2)$  angle is  $113.2^\circ$  and the average S-N bond length is 1.680 Å. The two morpholino groups of a molecule are slightly twisted; the torsion angles NSNC being about  $-67$  and  $+74^\circ$  for each morpholino ring.

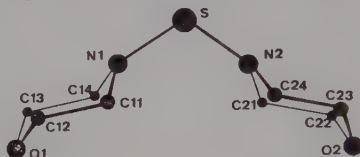


Fig. 1.  $\text{C}_8\text{H}_{16}\text{N}_2\text{O}_2\text{S}$  as seen normal to the NSN plane.

## 2-METHYL-4H-BENZOXAZIN-4-ONE (ACETYL ANTHRANYL)



M.C. ETTER, L.A. ERREDE and J. VICENS, 1982. Cryst. Struct. Comm., 11, 885-888.

Monoclinic,  $P2_1/c$ ,  $a = 7.353$ ,  $b = 10.398$ ,  $c = 10.432$  Å,  $\beta = 104.5$ ,  $Z = 4$ . Cu radiation,  $R = 0.086$  for 1014 reflexions.

The analysis was undertaken to gain insight into the mechanism of hydrolysis of the crystals of acetyl anthranyl. The packing pattern of acetyl anthranyl is shown in stereoview in Fig. 1. There are no unusually short intermolecular contacts. The carbonyl groups which are related by a screw axis form hydrophilic planes parallel to (001). Inversion related molecules form stacks parallel to the  $a$ -axis, which is also the long morphological axis of the crystal. When crystal hydrolysis takes place, reaction begins at the crystal ends ((100) face) and moves in the  $a$  direction. This would be consistent with water penetrating through the hydrophilic planes of the structure.

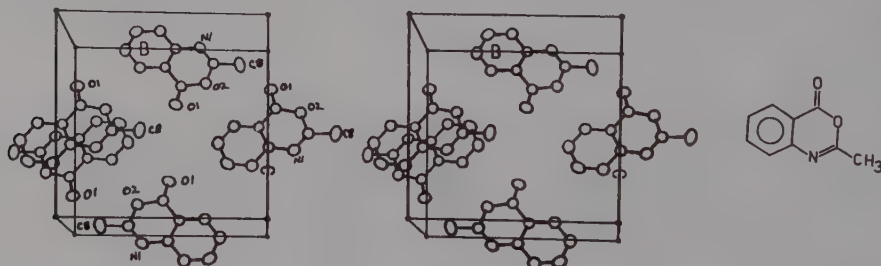
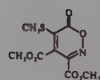
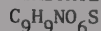


Fig. 1. Crystal packing of acetyl anthranyl.

## DIMETHYL 5-METHYLTHIO-6-OXO-6H-1,2-OXAZINE-3,4-DICARBOXYLATE



H. GOTTHARDT, O.M. HUSS and C.M. WEISSHUHN, 1982. Acta Cryst., B38, 680-682.

Monoclinic,  $P2_1/c$ ,  $a = 14.933$ ,  $b = 9.265$ ,  $c = 8.425$  Å,  $\beta = 92.03^\circ$ ,  $D_m = 1.42$ ,  $Z = 4$ . Mo radiation,  $R = 0.036$  for 2122 reflexions.

There are no unusual bond distances or angles in the molecule e.g. N-O 1.397(2), S-C(sp<sup>2</sup>) 1.746(1), S-C(sp<sup>3</sup>) 1.805(2), C=N 1.294(2) Å.

cis-4,5-TETRAMETHYLENE-3-METHYL-3,4,5,6-TETRAHYDRO-1,3-OXAZIN-2-THIONE  
C<sub>9</sub>H<sub>15</sub>NOS

A. KAPOR, B. RIBAR, A. KÁLMÁN, G. ARGAY, G. STÁJER and G. BERNÁTH, 1982. Cryst. Struct. Comm., 11, 1777-1780.

Monoclinic,  $P2_1/c$ ,  $a = 8.619$ ,  $b = 8.879$ ,  $c = 12.897$  Å,  $\beta = 100.99^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.066$  for 1148 reflexions.

The substituted N atom (Fig. 1) has the unexpected equatorial position. Bond lengths and angles are as anticipated, e.g. S-C(2) 1.677(6), C(2)-O(1) 1.335(7), C(2)-N(3) 1.329(7) Å.

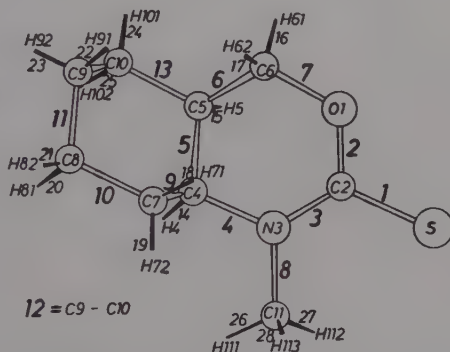


Fig. 1. A view of C<sub>9</sub>H<sub>15</sub>NOS.

2-AMINO-6-ETHYL-4,5,7,8-TETRAHYDRO-6H-OXAZOLO[5,4-d]AZEPINE  
C<sub>9</sub>H<sub>15</sub>N<sub>3</sub>O

A. CARPY, J.-M. LÉGER and J.-C. COLLETER, 1982. Cryst. Struct. Comm., 11, 53-58.

Monoclinic,  $P2_1/c$ ,  $a = 10.948$ ,  $b = 6.891$ ,  $c = 13.327$  Å,  $\beta = 85.79^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.042$  for 1204 reflexions.

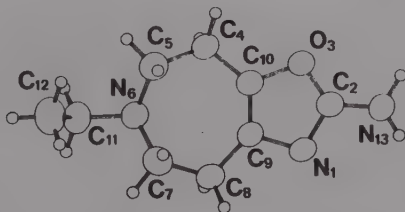
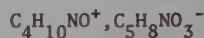
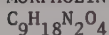


Fig. 1. The C<sub>9</sub>H<sub>15</sub>N<sub>3</sub>O molecule.

In this molecule (Fig. 1) the short N(1)-C(2) bond (1.299(3) Å) shows that the double bond is endocyclic; this is confirmed by the location of two H atoms on N(13). However C(2)-N(13) (1.335(3) Å) is shortened (partial double bond). The C(2)-O(3) bond is also shortened (1.360(3) Å) compared with O(3)-C(10) (1.407(3) Å). These two results indicate that a delocalization of the  $\pi$  electrons occurs. Therefore C(2) must be considered as the focus of the  $\pi$  electrons. The other bond lengths have expected values. The seven-membered ring adopts a chair conformation: atoms C(4), C(5), C(7) and C(8) being in a same plane, C(9) and C(10) are 0.534(2) and 0.495(2) Å respectively above the plane and N(6) is 0.775(2) Å below the plane.

# MORPHOLINIUM MORPHOLINOFORMATE



C.J. BROWN and L.R. GRAY, 1982. *Acta Cryst.*, **B38**, 2307-2308.

Triclinic,  $\text{P}\bar{1}$ ,  $a = 10.497$ ,  $b = 9.366$ ,  $c = 6.365$  Å,  $\alpha = 94.07$ ,  $\beta = 66.56$ ,  $\gamma = 106.47^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.060$  for 1473 reflexions.

The structure (Fig. 1) consists of two morpholinium morpholinoformate ion-pairs linked by N-H...O hydrogen bonds to form a discrete unit about an inversion centre.

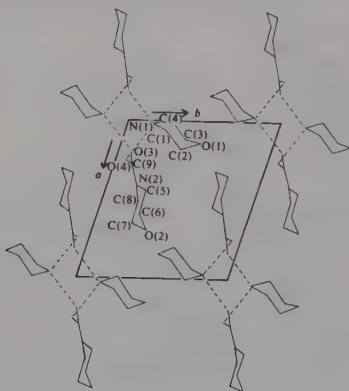


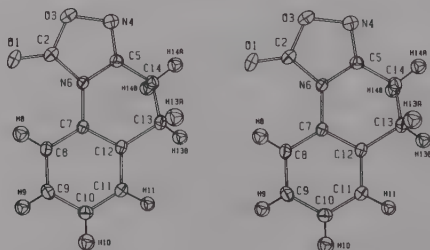
Fig. 1.  $\text{C}_4\text{H}_{10}\text{NO}^+, \text{C}_5\text{H}_8\text{NO}_3^-$ : packing arrangement in the unit cell.

# 4,5-DIHYDRO-1-OXO-1H-[1,2,4]OXADIAZOLO[4.3-a]QUINOLINE



M.Y. CHENG, H.O. LARSON and K. SEFF, 1982. *Acta Cryst.*, **B38**, 1335-1337.

Triclinic,  $\text{P}\bar{1}$ ,  $a = 8.008$ ,  $b = 8.862$ ,  $c = 7.848$  Å,  $\alpha = 92.11$ ,  $\beta = 118.93$ ,  $\gamma = 63.52^\circ$ ,  $D_m = 1.46$ ,  $Z = 2$ . Cu radiation,  $R = 0.033$  for 987 reflexions.



In the molecule (Fig. 1) the five-membered oxadiazolo ring is planar, C(13) and C(14) lie on opposite sides of the best plane of the heterocyclic six-membered ring, and the average bond length in the benzene ring, 1.383 Å, is normal.

#### SULPHAMETHOXAZOLE (Two Polymorphs)



G.P. BETTINETTI, F. GIORDANO, A. LA MANNA, G. GIUSEPPEPPI and C. TADINI, 1982. Cryst. Struct. Comm., 11, 821-828.

I. Monoclinic, C2/c,  $a = 16.062$ ,  $b = 5.479$ ,  $c = 25.757$  Å,  $\beta = 96.12^\circ$ ,  $D_m = 1.50$ ,  $Z = 8$ . Cu radiation,  $R = 0.032$  for 1330 reflexions.

II. Monoclinic, C2/c,  $a = 25.095$ ,  $b = 7.226$ ,  $c = 14.848$  Å,  $\beta = 117.99^\circ$ ,  $D_m = 1.44$ ,  $Z = 8$ . Cu radiation,  $R = 0.030$  for 1173 reflexions.

The isoxazole ring is planar in both polymorphs and the compound is present as the amido tautomeric form. In form I (Fig. 1) molecules are linked by weak N-H...O hydrogen bonds (3.24 Å), whereas in (II) dimers are formed via N-H...N hydrogen bonds (2.94 Å).

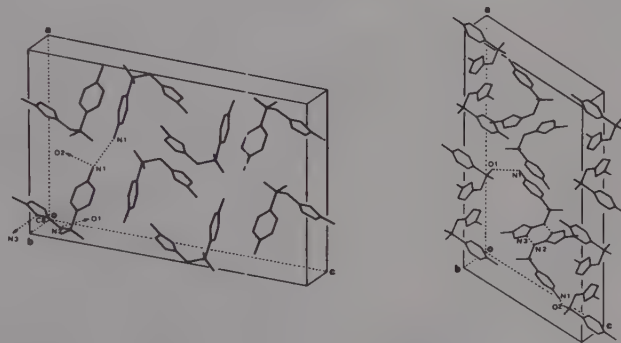
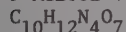


Fig. 1. The two forms of sulphamethazole (I) left and (II) right.

#### 9-ACETYL-4,5-(1-METHOXYETHYLIDENEDIOXY)-4,5-DIHYDROURIC ACID



M. POJE, B. ROČIĆ, I. VICKOVIĆ and M. BRUVO, 1982. J. Chem. Soc. Chem. Commun., 1338.

Monoclinic, P2<sub>1</sub>/c,  $a = 10.738$ ,  $b = 9.000$ ,  $c = 13.023$  Å,  $\beta = 91.54^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.043$  for 1116 reflexions.

The five- and six-membered N-containing rings are planar and the dioxalan ring has an envelope conformation (Fig. 1). Dihedral angles are: C<sub>3</sub>N<sub>2</sub>/C<sub>4</sub>N<sub>2</sub> 124.5, C<sub>3</sub>N<sub>2</sub>/C<sub>3</sub>O<sub>2</sub> 116.7 and C<sub>3</sub>O<sub>2</sub>/C<sub>4</sub>N<sub>2</sub> 118.8°. The molecular dimensions are normal. Molecules in the crystal are held together by hydrogen bonds: N(3)...O(9) 2.738(5), N(1)...O(6) 2.894(4) and N(7)...O(2) 2.802(3) Å.

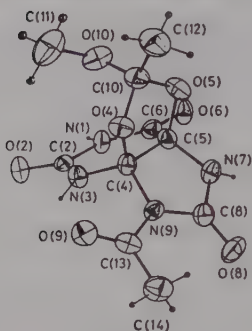
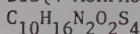


Fig. 1.  $C_{10}H_{12}N_4O_7$ : a view of the molecule.

BIS(4-MORPHOLINETHIOCARBONYL)DISULPHIDE



G.C. ROUT, M. SESHASAYEE and G. ARAVAMUDAN, 1982. Cryst. Struct. Comm., **11**, 1389-1393.

Orthorhombic,  $Pbca$ ,  $a = 9.257$ ,  $b = 14.949$ ,  $c = 21.114 \text{ \AA}$ ,  $Z = 8$ . Cu radiation,  $R = 0.064$  for 760 reflexions.

The morpholine rings have chair conformation (Fig. 1). The C-S-S-C torsion angle is  $99^\circ$ , with S-S 2.009(3), S(2)-C(1) 1.787(16), S(3)-C(2) 1.769(15), S(4)-C(2) 1.669(14), S(1)-C(1) 1.675(15) Å.

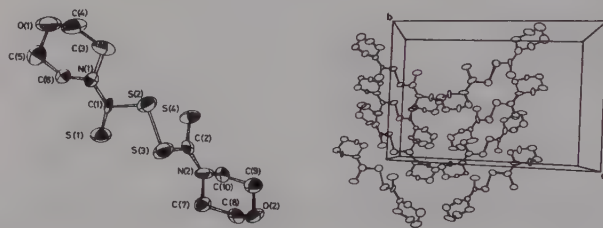


Fig. 1. Views of  $C_{10}H_{16}N_2O_2S_4$  and its packing.

METHYL 4-NITRO-3-PHENYLISOXAZOLE-5-CARBOXYLATE



S.B. BAUTZ, N.F. BLOM, J.C.A. BOYENS and J.P. MICHAEL, 1982. S. Afr. J. Chem., **35**, 166-170.

Orthorhombic,  $Pnma$ ,  $a = 20.121$ ,  $b = 6.738$ ,  $c = 8.269 \text{ \AA}$ ,  $D_m = 1.46$ ,  $Z = 4$ . Mo radiation,  $R = 0.086$  for 1214 reflexions.

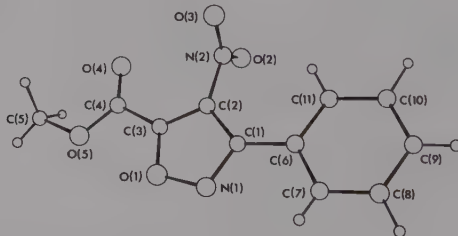


Fig. 1. The  $C_{11}H_8N_2O_5$  molecule.

The analysis identified this reaction by-product. The molecule (Fig. 1) has crystallographic mirror symmetry. The N(1)-O(1) distance is 1.403(5) Å.

3,3'-DIACETYL-4'-CHLORO-5-(TRICHLOROMETHYL)[4,5'-BIOXAZOLIDINYL]-2,2'-DIONE  
 $C_{11}H_{10}Cl_4N_2O_6$

T. KUNIEDA, Y. ABE, Y. IITAKA and M. HIROBE, 1982. J. Org. Chem., 47, 4291-4297.

Triclinic,  $P\bar{1}$ ,  $a = 14.510$ ,  $b = 10.748$ ,  $c = 10.575$  Å,  $\alpha = 99.82$ ,  $\beta = 98.12$ ,  $\gamma = 82.82^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.107$  for 4892 reflexions.

There are two independent molecules in the asymmetric unit; one of these is shown in Fig. 1. The other is similar. Molecular dimensions are normal.

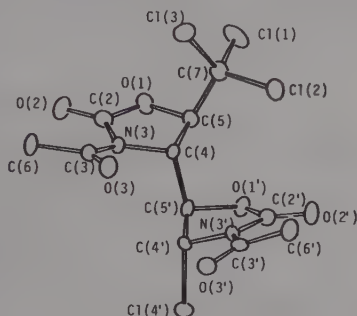


Fig. 1. A view of one of the  $C_{11}H_{10}Cl_4N_2O_6$  molecules.

#### SULPHISOXAZOLE

$C_{11}H_{13}N_3O_3S$

C.H. KOO, H.S. SHIN and S.I. CHO, 1982. Arch. Pharm. Res., 5, 79-86.

Orthorhombic,  $Pbca$ ,  $a = 14.492$ ,  $b = 11.563$ ,  $c = 14.900$  Å,  $D_m = 1.40$ ,  $Z = 8$ . Cu radiation,  $R = 0.094$  for 1360 reflexions. [See also the following Report.]

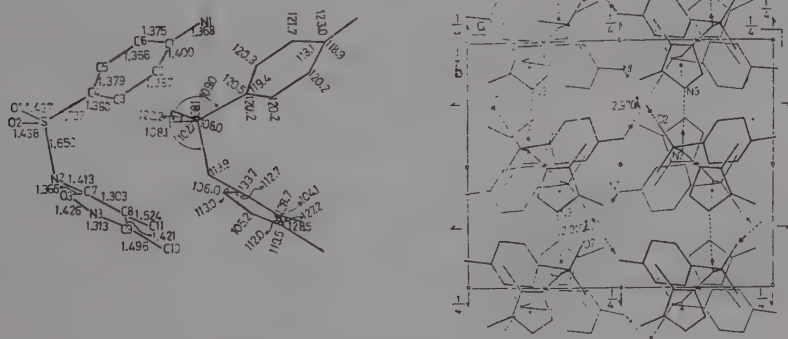


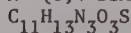
Fig. 1. Molecular structure of  $C_{11}H_{13}N_3O_3S$  showing bond lengths (Å) and angles ( $^\circ$ ) (left), and packing diagram viewed down the  $b$  axis (right).

The benzene ring plane makes an angle of  $68^\circ$  with the plane of the isoxazole ring, which is planar (Fig. 1). The conformational angle formed by the torsional angle C(4)-S-N(2)-C(7) is  $54^\circ$ . There are two intermolecular hydrogen bonds in the



structure; one is N-H...O with N...O 2.970 Å and the other is N-H...N with N...N 2.915 Å. A two-dimensional network of hydrogen bonds forms infinite molecular sheets parallel to (001) plane, and adjacent sheets are bound together by van der Waals forces.

N<sup>1</sup>-(3,4-DIMETHYL-5-ISOXAZOLYL)SULPHANILAMIDE (SULPHISOXAZOLE)



C. CHATTERJEE, J.K. DATTAGUPTA and N.N. SAHA, 1982. *Acta Cryst.*, **B38**, 1845-1847.

Orthorhombic, *Pbca*, *a* = 14.917, *b* = 14.506, *c* = 11.570 Å, *D<sub>m</sub>*<sup>-</sup> = 1.402, *Z* = 8. Cu radiation, *R* = 0.069 for 1765 reflexions. [See also the preceding Report.]

The molecule has normal molecular geometry e.g. S-O(1) 1.428(4), S-O(2) 1.433(4), S-N(2) 1.653(3) Å, O(1)-S-O(2) 119.8° (Fig. 1). The structure is stabilised by N(1)-H...O(2') and N(2)-H...N(3) bonds of length 2.990(6) and 2.943(5) Å respectively.

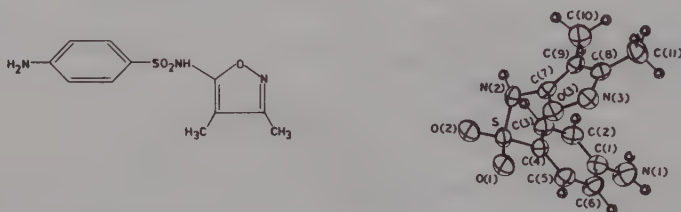
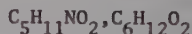
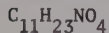


Fig. 1. The sulphisoazole molecule.

N-METHYLMORPHOLINE N-OXIDE trans-1,2-CYCLOHEXANEDIOL



H. CHANZY, E. MAIA and S. PÉREZ, 1982. *Acta Cryst.*, **B38**, 852-855.

Monoclinic, *P2<sub>1</sub>/c*, *a* = 6.137, *b* = 10.153, *c* = 21.015 Å, *β* = 94.33°, *D<sub>m</sub>* = 1.19, *Z* = 4. Cu radiation, *R* = 0.042 for 1019 reflexions.

The morpholine ring (Fig. 1) takes the chair form with axial N-O 1.383(3) Å. In the trans-1,2-cyclohexanediol molecule a distinct shrinkage of the bond between the two oxygen-bearing C atoms is observed (1.473 Å). The molecule is in a chair conformation with both hydroxylic O atoms equatorial. The N-O structuring site links, through hydrogen bonds, contiguous trans-1,2-cyclohexanediol molecules oriented along the *a* axis.

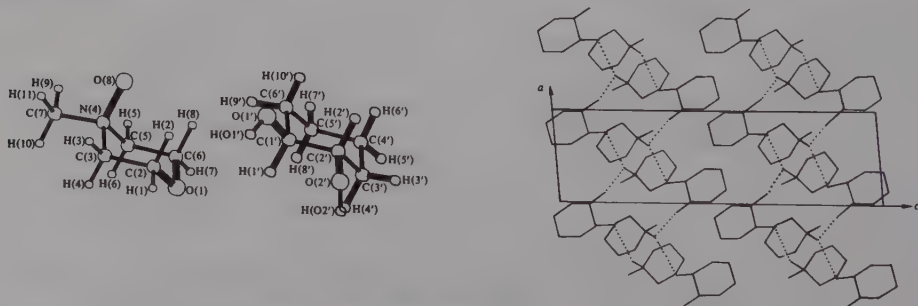


Fig. 1.  $\text{C}_5\text{H}_{11}\text{NO}_2 \cdot \text{C}_6\text{H}_{12}\text{O}_2$ : view of the molecules of N-methylmorpholine N-oxide and trans-1,2-cyclohexanediol (left) and a projection of the structure down the *b* axis (right); hydrogen bonds are shown as dashed lines.

5-CARBAMOYL-4-(METHYLTHIO)-2-PHENYL-1,3-OXAZINE-6-THIONE  
 $C_{12}H_{10}N_2O_2S_2$  (I)

5-CARBAMOYL-4-(METHYLTHIO)-2-PHENYL-1,3-THIAZIN-6-ONE  
 $C_{12}H_{10}N_2O_2S_2$  (II)

5-CARBAMOYL-4-(METHYLTHIO)-2-PHENYL-2,3-DIHYDRO-1,3-THIAZIN-6-ONE  
 $C_{12}H_{12}N_2O_2S_2$  (III)

M. YOKOYAMA, M. NAKAMURA, H. OHTEKI, T. IMAMOTO and K.-I. YAMAGUCHI, 1982. J. Org. Chem., 47, 1090-1094.

I. Monoclinic,  $P2_1/c$ ,  $a = 5.049$ ,  $b = 8.008$ ,  $c = 33.128$  Å,  $\beta = 112.34^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.093$  for 1990 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 7.688$ ,  $b = 5.156$ ,  $c = 31.473$  Å,  $\beta = 105.52^\circ$ ,  $Z = 4$ . Cu radiation.

III. Orthorhombic,  $Pbcn$ ,  $a = 13.218$ ,  $b = 9.562$ ,  $c = 20.220$  Å,  $Z = 8$ . Mo radiation,  $R = 0.062$  for 2598 reflexions.

The analyses established the structures shown above.

3-[o-(CARBOMETHOXY)PHENYL]-5-(HYDROXYMETHYL)-6H-1,4,2-DIOXAZINE  
 $C_{12}H_{13}NO_5$

N. AMLAIKY, G. LECLERC and A. CARPY, 1982. J. Org. Chem., 47, 517-523.

Triclinic,  $P1$ ,  $a = 7.638$ ,  $b = 9.151$ ,  $c = 9.624$  Å,  $\alpha = 84.68^\circ$ ,  $\beta = 70.35^\circ$ ,  $\gamma = 68.04^\circ$ ,  $Z = 1$ . Cu radiation,  $R = 0.079$  for 1731 reflexions.

Treatment of N-[(2,3-epoxypropyl)oxy]phthalimide with alcohols was shown to give the dioxazine product, Fig. 1, by X-ray analysis. The dioxazine ring has four coplanar atoms, C(7), O(8), N(12) and O(11), with C(9) 0.27 Å below and C(10) 0.37 Å above this plane. Hydrogen bonds link the molecules in chains.

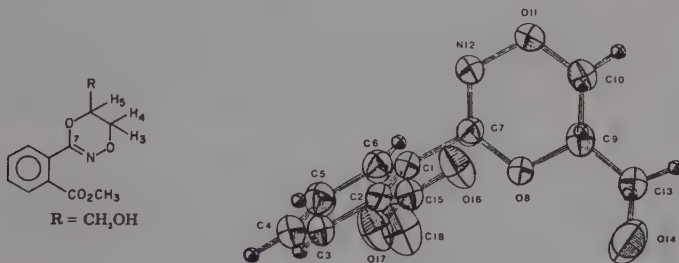


Fig. 1. Formula and view of the dioxazine molecule,  $C_{12}H_{13}NO_5$ .

9,10-BENZO-1,7-DIAZA-4-OXACYCLOUNDECANE-8,11-DIONE  
 $C_{12}H_{14}N_2O_3$  (I)

12,13-BENZO-1,10-DIAZA-4,7-DIOXACYCLOTETRADECANE-11,14-DIONE  
 $C_{14}H_{18}N_2O_4$  (II)

Yu.G. GANIN, E.V. GANIN, Yu.A. SIMONOV, V.F. ANIKIN and G.L. KAMALOV, 1982. Zh. Strukt. Khim., 23-6, 103-107 [J. Struct. Chem., 23, 909-913].

I. Monoclinic,  $P2_1/n$ ,  $a = 14.187$ ,  $b = 10.909$ ,  $c = 7.619$  Å,  $\gamma = 101.66^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.054$  for 1487 reflexions.

II. Orthorhombic,  $Pbca$ ,  $a = 19.600$ ,  $b = 16.639$ ,  $c = 8.890$  Å,  $Z = 8$ . Cu radiation,  $R = 0.079$  for 2250 reflexions.

The phthalic acid fragments in the 11-membered ring in I and in the 14-membered ring in II have different structures (Fig. 1). Intra-annular torsion angles are also shown in Fig. 1. Bond lengths and angles ( $\sigma = 0.004$ - $0.006$  Å and  $0.2$ - $0.3^\circ$  for I,  $0.005$ - $0.007$  Å and  $0.3$ - $0.4^\circ$  for II) are normal. Non-bonded 1-4 interactions involving carbonyl groups are discussed.

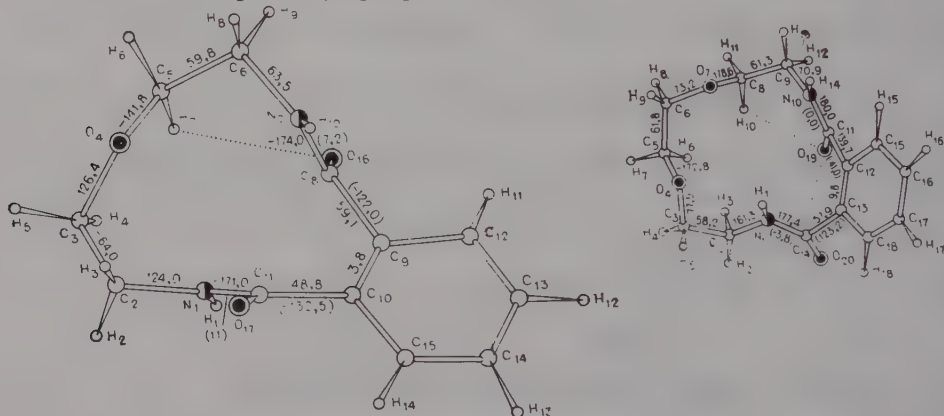


Fig. 1. The molecules  $C_{12}H_{14}N_2O_3$  (left) and  $C_{14}H_{18}N_2O_4$  (right).

5-METHOXYMETHYL-3-(3-METHYLPHENYL)-2-OXAZOLIDINONE  
 $C_{12}H_{15}NO_3$

F. DURANT, F. LEFEVRE, G. EVRARD and A. MICHEL, 1982. Cryst. Struct. Comm., **11**, 975-981.

Monoclinic,  $P2_1$ ,  $a = 8.665$ ,  $b = 6.341$ ,  $c = 10.434$  Å,  $\beta = 98.39^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.05$  for 1052 reflexions.

The oxazolidinone ring (Fig. 1) is quasi-planar and nearly coplanar with the adjacent phenyl ring. The dimensions are in accord with a delocalization scheme as shown in the figure.

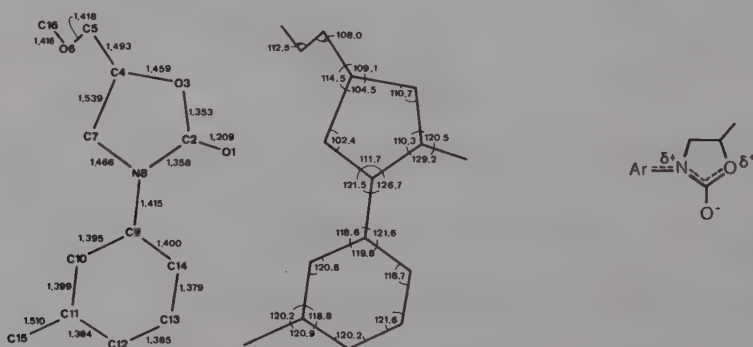


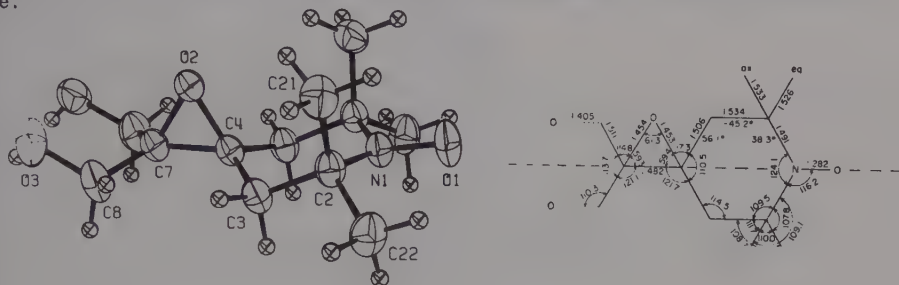
Fig. 1. Bond lengths and angles in  $C_{12}H_{15}NO_3$ .

4,7-OXIDO-7-METHYL-7-HYDROXYMETHYL-2,2,6,6-TETRAMETHYLPIPERIDIN-1-OXYL  
 $C_{12}H_{22}NO_3$

M. CYGLER, 1982. *Canad. J. Chem.*, **60**, 2392-2397.

Monoclinic,  $P2_1/m$ ,  $a = 8.041$ ,  $b = 13.348$ ,  $c = 5.945$  Å,  $\beta = 95.90^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.039$  for 1304 reflexions.

The molecule lies on a crystallographic mirror plane and the terminal  $CH_3$ - and  $-CH_2OH$  groups are mutually disordered. The piperidine ring (Fig. 1) has a chair conformation. The N-O bond is  $1.282(2)$  Å and is inclined at  $18.5(2)^\circ$  to the CNC plane.



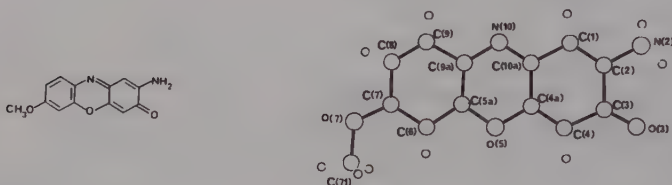
## 2-AMINO-7-METHOXY-3H-PHENOXAZIN-3-ONE

 $C_{13}H_{10}N_2O_3$ 

R.G. BUCKLEY, J. CHARALAMBOUS and K. HENRICK, 1982. Acta Cryst., B38, 289-291.

Monoclinic,  $P2_1/n$ ,  $a = 21.976$ ,  $b = 3.901$ ,  $c = 12.523$  Å,  $\beta = 95.68^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.052$  for 598 reflexions.

Bond lengths within the quinoidal portion of the almost planar phenoxazinone ring (Fig. 1) show the pattern of bond lengths corresponding to the Kekulé formula shown. Bond lengths in the phenyl ring are normal, with mean C-C 1.391 Å.

Fig. 1. Perspective view of the  $C_{13}H_{10}N_2O_3$  molecule.

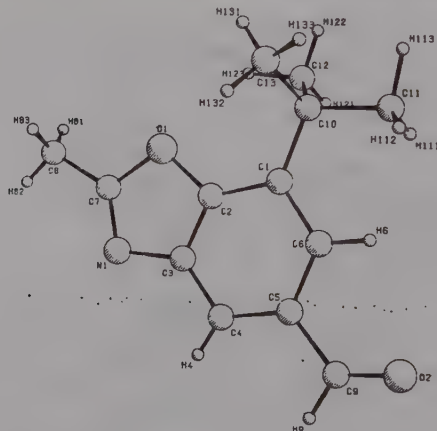
## 7-t-BUTYL-2-METHYL-5-BENZOXAZOLECARBALDEHYDE

 $C_{13}H_{15}NO_2$ 

E.-L. DREHER, J. BRACHT, M. EL-MOBAYED, P. HUTTER, W. WINTER and A. RIEKER, 1982. Chem. Ber., 115, 288-308.

Monoclinic,  $P2_1$ ,  $a = 7.688$ ,  $b = 10.117$ ,  $c = 8.507$  Å,  $\beta = 116.88^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.050$  for 778 reflexions.

Except for C(12) and C(13) of the t-butyl group the molecule (Fig. 1) is planar within experimental error. Bond lengths and angles have expected values.

Fig. 1. The structure of  $C_{13}H_{15}NO_2$ .

## 5-NITRO-2-CYCLOHEXYLAMINO BENZOXAZOLE

 $C_{13}H_{15}N_3O_3$

V.B. RYBAKOV, V.M. IONOV, K.A. PASESHNICHENKO and K.D. ATANASOV, 1982. Zh. Strukt. Khim., 23-4, 163-165 [J. Struct. Chem., 23, 634-636].

Monoclinic,  $P2_1/b$ ,  $a = 8.552$ ,  $b = 9.488$ ,  $c = 16.011$  Å,  $\gamma = 101.25^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.053$  for 1239 reflexions.

The molecule (Fig. 1) exhibits a planar fused-ring system, the nitro group being rotated  $12.2^\circ$  out of the plane. The cyclohexane ring has a chain conformation. Bond lengths ( $\sigma = 0.008$  Å) are normal.

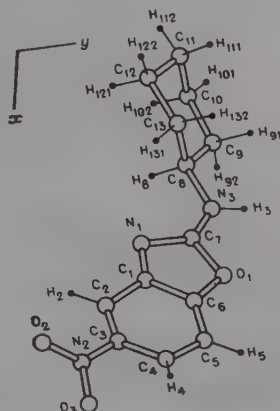


Fig. 1. The  $C_{13}H_{15}N_3O_3$  molecule.

#### 4-AMIDINOPHENYLACETIC ACID MORPHOLIDE HYDROIODIDE

$C_{13}H_{18}IN_3O_2$

G. RECK, V. HAGEN and G. WAGNER, 1982. Cryst. Struct. Comm., 11, 1099-1105.

Monoclinic,  $P2_1/n$ ,  $a = 11.234$ ,  $b = 13.343$ ,  $c = 10.056$  Å,  $\beta = 97.98^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.049$  for 3341 reflexions.

The amidine plane is inclined at  $36.7^\circ$  to the phenyl plane (Fig. 1) and C(4)-C(15) (1.473(7) Å) has partial double bond character. The amidine  $NH_2$  groups also participate in N-H...I hydrogen bonding. The morpholine ring has a chair conformation.

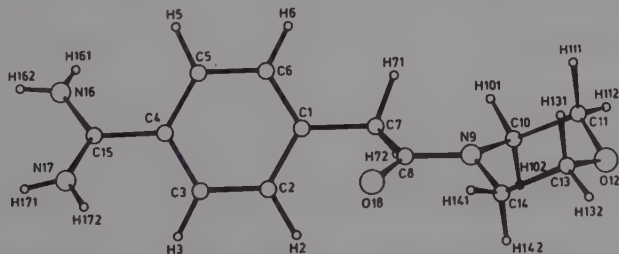


Fig. 1. The  $C_{13}H_{18}N_3O_2^+$  ion.

#### 3,4-BIS(4-CHLOROPHENYL)SYDNONE

$C_{14}H_8Cl_2N_2O_2$

J.Z. GOUGOUTAS, W.H. OJALA and M. MALLEY, 1982. Cryst. Struct. Comm., 11, 1731-1736.



Monoclinic,  $P2_1/c$ ,  $a = 14.176$ ,  $b = 6.925$ ,  $c = 13.894$  Å,  $\beta = 95.09^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.044$  for 1100 reflexions.

In the five-membered sydnone ring (Fig. 1) the C(11),N(7),N(8),P(9) fragment is planar and C(10) is displaced 0.029 Å from this plane. The phenyl rings are inclined in the same sense with respect to the sydnone ring (torsion angles C(3)-C(4)-N(7)-C(11)  $-52.3^\circ$ , C(13)-C(12)-C(11)-N(7)  $-33.2^\circ$ ).

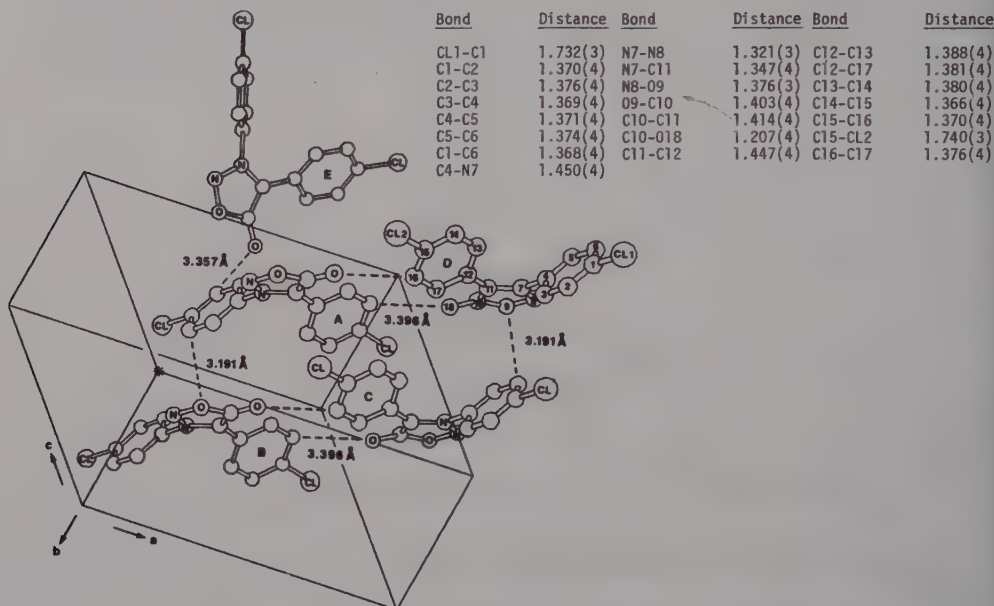


Fig. 1. The packing of  $C_{14}H_8Cl_2N_2O_2$ , and bond lengths.

3a,4-DIHYDRO-1-METHYL-3a-PHENYL-1,4-METHANO-5H-OXAZOLO[3,2-a]QUINOLIN-2(1H)-ONE  
 $C_{14}H_{15}NO_2$



A. PADWA, H.L. GINGRICH and R. LIM, 1982. J. Org. Chem., 47, 2447-2456.

Monoclinic,  $P2_1/n$ ,  $a = 10.522$ ,  $b = 8.858$ ,  $c = 16.5818$  Å,  $\beta = 94.46^\circ$ ,  $Z = 4$ .  $R = 0.051$ .

X-ray analysis established the structure shown above ( $R = Ph$ ).

2-PHENYL-2-METHYL-cis-5,6-TRIMETHYLENE-2,3,5,6-TETRAHYDRO-1,3-OXAZIN-4-ONE  
 $C_{14}H_{17}NO_2$

G. ARGAY, A. KÁLMÁN, B. RIBÁR, F. FÜLÖP and G. BERNÁTH, 1982. Cryst. Struct. Comm., 11, 959-964.

Monoclinic,  $P2_1/c$ ,  $a = 18.021$ ,  $b = 7.915$ ,  $c = 17.851$  Å,  $\beta = 101.27^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.064$  for 2981 reflexions.

In the asymmetric unit there are two molecules bound together by hydrogen bonds via N3-H3...O27 (N...O 2.894(5) Å) and N20-H20...O10 (N...O 2.823(5) Å) pairs (Fig. 1). The conformation of the symmetry independent molecules A and B differs visibly only in the puckering of the cyclopentane rings. While the puckering

amplitude  $Q$  is practically identical ( $0.33 \text{ \AA}$  for molecule A and  $0.34 \text{ \AA}$  for molecule B),  $\phi$  is  $170.4^\circ$  for molecule A and  $154.2^\circ$  for molecule B.

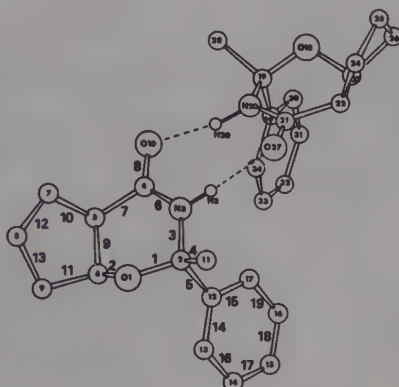


Fig. 1. A perspective view of the hydrogen-bonded molecules in  $C_{14}H_{17}NO_2$ .

2-(p-NITROPHENYL)-cis-5,6-TETRAMETHYLENE-2,3,5,6-TETRAHYDRO-1,3-OXAZINE  
 $C_{14}H_{18}N_2O_3$  (I)

2-(p-NITROPHENYL)-cis-4,5-TETRAMETHYLENE-2,3,4,5-TETRAHYDRO-1,3-OXAZINE  
 $C_{14}H_{18}N_2O_3$  (II)

Gy. ARGAY, A. KÁLMÁN, F. FÜLÖP and G. BERNÁTH, 1982. Acta Chim. Acad. Sci. Hung., 109, 39-49.

I. Monoclinic,  $P2_1/c$ ,  $a = 6.856$ ,  $b = 21.063$ ,  $c = 10.449 \text{ \AA}$ ,  $\beta = 117.82^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.033$  for 1730 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 6.585$ ,  $b = 12.507$ ,  $c = 16.054 \text{ \AA}$ ,  $\beta = 91.61^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.076$  for 1633 reflexions.

In both isomers the conformation of the hetero ring is an almost perfect chair form (Fig. 1). The hetero atoms (O(1) or N(3)) are axial while the methylene group bound to C(5) is equatorial. The phenyl rings assume synperiplanar positions relative to the O(1) atoms, and the para- $NO_2$  moiety lies in the plane of the phenyl ring in I while in II is twisted by  $8^\circ$  out of it.

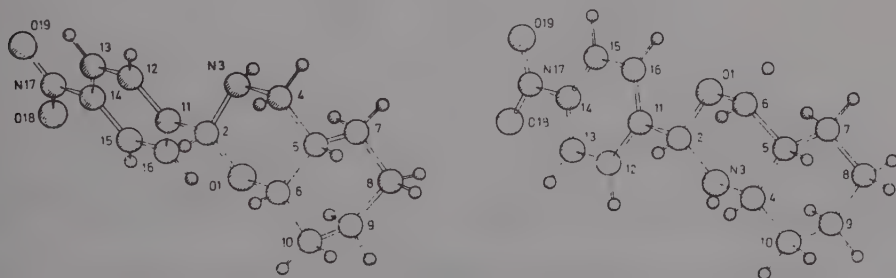


Fig. 1. Molecular structures of  $C_{14}H_{18}N_2O_3$  (I) (left) and II (right).

5-[(4-ALLYL-2-METHOXYPHENOXY)METHYL]-2-AMINO- $\Delta^2$ -1,3-OXAZOLINE  
 $C_{14}H_{18}N_2O_3$

C. JARRY, M. GOURSOLLE, J.M. LÉGER and A. CARPY, 1982. *Acta Cryst.*, **B38**, 964-966.

Triclinic,  $P\bar{1}$ ,  $a = 5.346$ ,  $b = 11.626$ ,  $c = 11.787$  Å,  $\alpha = 108.72$ ,  $\beta = 98.14$ ,  $\gamma = 91.40^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.047$  for 1059 reflexions.

Bond lengths in the molecule (Fig. 1) show that no delocalisation of electrons occurs between the oxazoline ring and the amine group. The molecules are linked into dimers by N-H...N hydrogen bonds with N...N 2.97 Å.

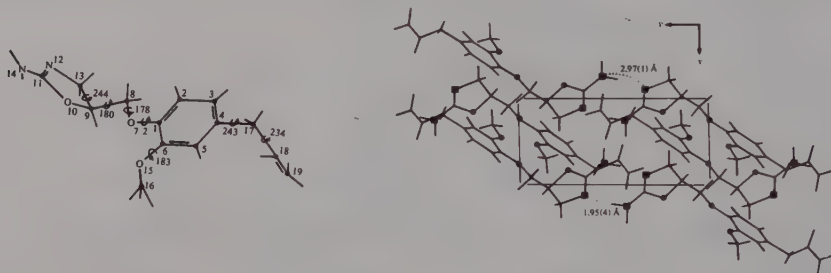


Fig. 1.  $C_{14}H_{18}N_2O_3$ : a view of the molecule showing torsion angles (left) and a projection of the structure on the (001) plane (right).

3-(N-(2'-N'-MORPHOLINO)ETHYLAMINO)-6-PHENYLPYRIDAZINE DIHYDROCHLORIDE DIHYDRATE  
 $C_{16}H_{22}Cl_2N_4O_4 \cdot 2H_2O$

A. MICHEL, R. GUSTIN, G. EVRARD and F. DURANT, 1982. *Bull. Soc. Chim. Belg.*, **91**, 49-55.

Triclinic,  $P\bar{1}$ ,  $a = 11.112$ ,  $b = 13.549$ ,  $c = 6.790$  Å,  $\alpha = 96.43$ ,  $\beta = 94.04$ ,  $\gamma = 114.99^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.05$  for 2937 reflexions.

In the molecule (Fig. 1), the morpholine nitrogen (N4) and N15 of the pyridazine are protonated. The molecular dimensions are consistent with contributions from canonical forms shown in Fig. 1. In the crystal structure the ions and water molecules are linked by hydrogen bonds.

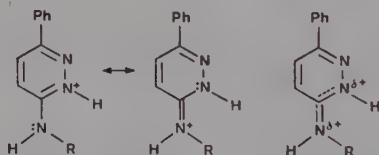


Fig. 1. Canonical forms for the cation in  $C_{16}H_{22}Cl_2N_4O_4 \cdot 2H_2O$ .

1,7,10,16-TETRAOXA-4,13-DIAZACYCLOOCTADECANE THIIOUREA (1:4 ADDUCT)

$C_{16}H_{42}N_{10}O_4S_4$

$C_{12}H_{26}N_2O_4 \cdot 4(CH_4N_2S)$

G. WEBER, 1982. *Acta Cryst.*, **B38**, 2712-2715.

Triclinic,  $P\bar{1}$ ,  $a = 9.121$ ,  $b = 9.849$ ,  $c = 9.883$  Å,  $\alpha = 117.07$ ,  $\beta = 109.12$ ,  $\gamma = 97.09^\circ$ ,  $Z = 1$ . Mo radiation,  $R = 0.036$  for 2314 reflexions.

In the complex (Fig. 1) two N-H...O and two N-H...N hydrogen bonds between the host and two guest molecules and two additional intra-annular N-H...O contacts give rise to the 'biangular' conformation of the macrocycle. The remaining thiourea molecules are involved in N-H...S bridges linking these units.

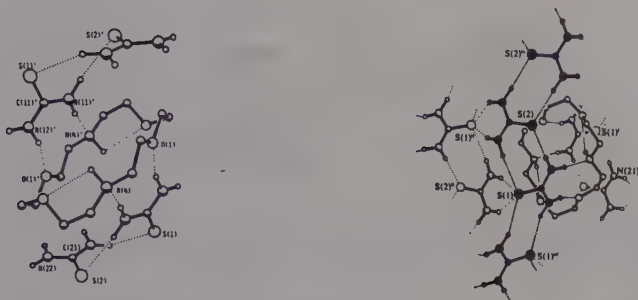


Fig. 1.  $C_{12}H_{26}N_2O_4 \cdot 4(CH_4N_2S)$ : views of the complex.

1,5-DIPHENYL-6-OXA-4-AZASPIRO[2.4]HEPT-4-EN-7-ONE  
 $C_{17}H_{13}NO_2$

S.W. KING, J.M. RIODAN, E.M. HOLT and C.H. STAMMER, 1982. *J. Org. Chem.*, **47**, 3270-3273.

Triclinic,  $P\bar{1}$ ,  $a = 5.484$ ,  $b = 11.101$ ,  $c = 11.189$  Å,  $\alpha = 85.62$ ,  $\beta = 82.39$ ,  $\gamma = 84.50^\circ$ ,  $D_m = 1.305$ ,  $Z = 2$ . [?] radiation,  $R = 0.069$  for 2031 reflexions.

The structure (Fig. 1) shows the five-membered azlactone ring (C4, O1, C5, C3, N1) to be flat (deviation from planarity averaged 0.009 Å) and to be close to being coplanar with the benzene ring (C6, C7, C8, C9, C10, C11) substituted on carbon 5 (angle between planes  $9.20^\circ$ ). The three-membered ring (C1, C2, C3), which includes C-4 of the oxazolone ring, subtends an angle of  $87.45^\circ$  with the plane of that ring and an angle of  $71.07^\circ$  with the plane of the benzene ring (C12, C13, C14, C15, C16, C17). The benzene ring and N1 are shown to be cis to each other with respect to the cyclopropane ring.

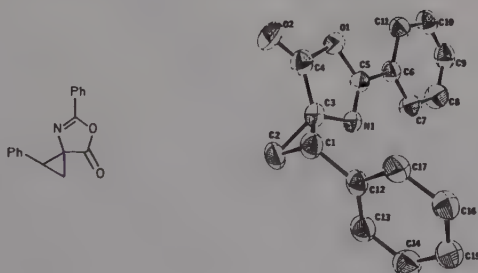


Fig. 1. The  $C_{17}H_{13}NO_2$  molecule.

(S,S)-(-)-2-(d- $\alpha$ -BROMO- $\pi$ -CAMPHORSULFONYL)-3-(2-CHLORO-5-NITROPHENYL)OXAZIRIDINE  
 $C_{17}H_{18}BrClN_2O_6S$

F.A. DAVIS, R.H. JENKINS, Jr., S.B. AWAD, O.D. STRINGER, W.H. WATSON and J. GALLOY, 1982. *J. Am. Chem. Soc.*, **104**, 5412-5418.

Orthorhombic,  $P2_12_12_1$ ,  $a = 8.109$ ,  $b = 33.395$ ,  $c = 7.368$  Å,  $Z = 4$ . Cu radiation,  $R = 0.052$  for 1544 reflexions (absolute configuration).

The molecule (Fig. 1) may be stabilized by intramolecular C-H...N and C-H...O interactions ( $H...N$  2.46(1),  $H...O < 2$  Å). The phenyl and oxaziridine rings make an angle of  $66.5(9)^\circ$ . The bond lengths in the three-membered ring are: N-O 1.48(1), N-C 1.42(1), and O-C 1.409(9) Å. Other dimensions are normal.

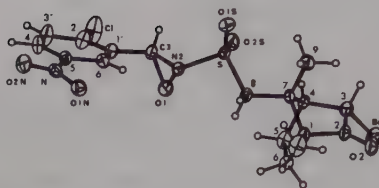


Fig. 1. Structure and absolute configuration of  $C_{17}H_{18}BrClN_2O_6S$ .

8,8-DIMETHYL-1,15-DIAZA-6,10-DIOXACYCLONONADECAN-2,5,11,14-TETRAONE  
 $C_{17}H_{28}N_2O_6$

M.M. BOTOSHANSKII, Yu.A. SIMONOV, V.K. BEL'SKII, N.G. LUK'YANENKO and V.A. SHAPKIN, 1982. *Kristallografiya*, 27, 1112-1115 [*Sov. Phys. Crystallogr.*, 27, 665-667].

Triclinic,  $P\bar{1}$ ,  $a = 9.888$ ,  $b = 10.296$ ,  $c = 11.041$  Å,  $\alpha = 66.07^\circ$ ,  $\beta = 91.38^\circ$ ,  $\gamma = 67.76^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.046$  for 1790 reflexions.

The nineteen-membered macro-ring (Fig. 1) is of Dale's conformation type [811]. The ester and amide groups are planar. The packing of the macro-rings in the crystal is mainly governed by N-H...O hydrogen bonds (intermolecular) which link the molecules along the  $a$  axis. The N...O distances are: 2.88 Å (N(2)...O(1)) and 2.90 Å (N(1)...O(4)).

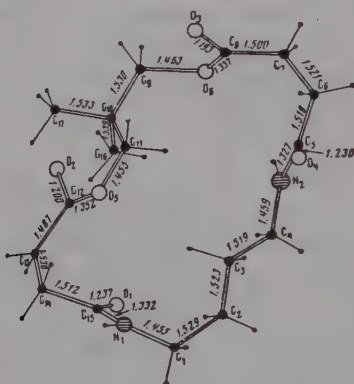
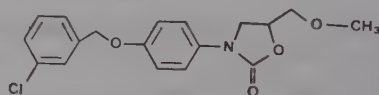


Fig. 1. Interatomic distances (Å) in the molecule of  $C_{17}H_{28}N_2O_6$ .

3-[4-[(3-CHLOROPHENYL)METHOXY]PHENYL]-5-METHOXY-2-OXAZOLIDINONE  
 $C_{18}H_{18}ClNO_4$

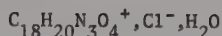


F. DURANT, F. LEFEVRE, G. EVRARD and A. MICHEL, 1982. *Bull. Soc. Chim. Belg.*, 91, 831-832.

Monoclinic,  $P2_1/c$ ,  $a = 14.424$ ,  $b = 5.998$ ,  $c = 20.071$  Å,  $\beta = 106.61^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.06$  for 1616 reflexions.

The dihedral angle between phenyl and adjacent oxazolidinone rings is  $7.2^\circ$ . Molecular dimensions are in accord with a resonance scheme involving delocalization throughout the Ph-N-CO<sub>2</sub> moiety.

## NITROXAZEPINE HYDROCHLORIDE MONOHYDRATE



R. USHA, M.M. BHADBADE, K. VENKATESAN, K. NAGARAJAN and J. DAVID, 1982. Acta Cryst., B38, 1854-1857.

Orthorhombic,  $\text{Pn}2_1\text{a}$ ,  $a = 7.710$ ,  $b = 11.455$ ,  $c = 21.199 \text{ \AA}$ ,  $D_m = 1.38$ ,  $Z = 4$ . Cu radiation,  $R = 0.045$  for 1470 reflexions.

Within the molecule (Fig. 1) the three-ring system is folded about the O(6),-N(13),C(14) plane; rings A and B make angles of  $143.5(5)$  and  $21.9(5)^\circ$  with this plane. The angle between rings A and B is  $125.0(5)^\circ$ . The central seven-membered ring has an approximately boat conformation. The C(20)-C(21)-C(22)-N(23) torsion angle is  $-58.9^\circ$  and N(23) is  $7.697(4)$  and  $6.755 \text{ \AA}$  from the centres of rings A and B. The structure is stabilised by N-H...Cl ( $3.038 \text{ \AA}$ ) and water (O)-H...Cl ( $3.302$  and  $3.309 \text{ \AA}$ ) hydrogen bonds.

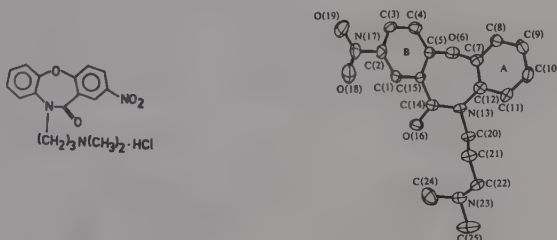


Fig. 1. The nitroxazepine molecule.

## 3-ETHYL-4,5-BIS(METHOXYCARBONYL)-2-PHENYL-2-VINYL-2,3-DIHYDRO-6H-1,3-OXAZINE



I. GOLDBERG, D. SAAD, E. SHALOM and S. SHATZMILLER, 1982. J. Org. Chem., 47, 2192-2194.

Monoclinic,  $\text{P}2_1/\text{n}$ ,  $a = 7.834$ ,  $b = 13.334$ ,  $c = 16.595 \text{ \AA}$ ,  $\beta = 95.06^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.039$  for 2074 reflexions.

The overall shape (Fig. 1) of the oxazine system is envelope-like, where the oxygen atom deviates by about  $0.5 \text{ \AA}$  from the mean plane defined by the remaining atoms. The enamine part is almost fully conjugated to the  $\beta$ -carbomethoxy group. This is reflected in a coplanar arrangement of the two fragments and the distribution of bond distances along the vinylogous urethan moiety. The second carbomethoxy group forms a torsion angle of  $72^\circ$  with the plane of the conjugated system and is characterized by usual geometry. Crystal forces appear to have little influence on the molecular geometry, as all intermolecular distances are greater than or approximately equal to the sums of the van der Waals radii.

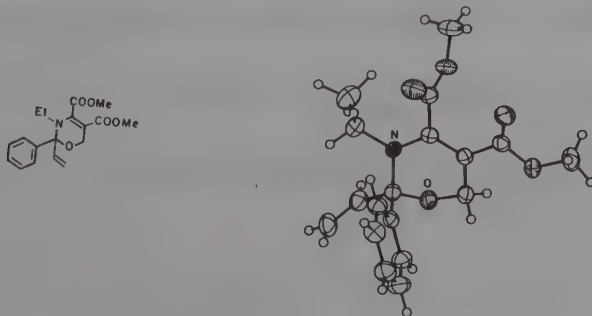


Fig. 1. The  $\text{C}_{18}\text{H}_{21}\text{NO}_5$  structure.



(2R, $\alpha$ S)-6(e)-t-BUTYL-2-( $\alpha$ -METHYLBENZYL)-1,2-OXAZASPIRO[2.5]OCTANE  
 $C_{18}H_{27}NO$  (I)

(5S, $\alpha$ S)-5-t-BUTYL-1-( $\alpha$ -METHYLBENZYL)HEXAHYDRO-2-AZEPINONE  
 $C_{18}H_{27}NO$  (II)

A. LATTES, E. OLIVEROS, M. RIVIÈRE, C. BELZECKI, D. MOSTOWICZ, W. ABRAMSKJ,  
 C. PICCINNI-LEOPARDI, C. GERMAIN and M. VAN MEERSSCHE, 1982. *J. Am. Chem. Soc.*, **104**,  
 3929-3934.

I. Orthorhombic,  $P2_12_12_1$ ,  $a = 17.741$ ,  $b = 6.130$ ,  $c = 15.558$  Å,  $Z = 4$ . Mo radiation,  
 $R = 0.061$  for 780 reflexions (absolute configuration).

II. Monoclinic,  $P2_1$ ,  $a = 10.767$ ,  $b = 6.584$ ,  $c = 11.666$  Å,  $\beta = 100.41^\circ$ ,  $Z = 2$ . Mo  
 radiation,  $R = 0.041$  for 1124 reflexions (absolute configuration).

The two molecules are shown in Fig. 1. The most interesting feature of I is  
 the oxaziridine ring with the following geometry: O-N 1.535(8), O-C 1.428(10),  
 N-C 1.456(11) Å, N-O-C 58.7(5), O-N-C 57.0(5), and O-C-N 64.3(6)°. Molecules of II  
 contain seven-membered lactam rings which adopt a chair conformation. Bond lengths  
 in II ( $\sigma = 0.004$ -0.008 Å) are normal.

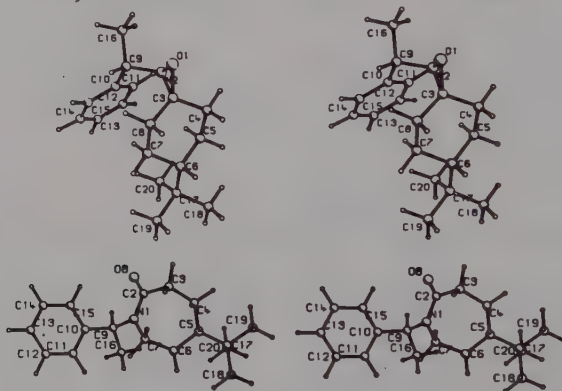
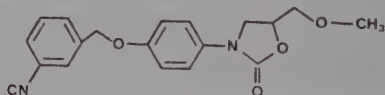


Fig. 1. Stereoviews of the  $C_{18}H_{27}NO$  molecules I (top) and II (bottom).

3-[4-[(3-CYANOPHENYL)METHOXY]PHENYL]-5-(METHOXYMETHYL)-2-OXAZOLIDINONE  
 $C_{19}H_{18}N_2O_4$



F. DURANT, F. LEFÈVRE, G. EVRARD and C. LAMOTTE, 1982. *Bull. Soc. Chim. Belg.*, **91**,  
 949-950.

Monoclinic,  $P2_1/c$ ,  $a = 12.940$ ,  $b = 6.064$ ,  $c = 21.031$  Å,  $\beta = 100.31^\circ$ ,  $Z = 4$ . Cu  
 radiation,  $R = 0.06$  for 1207 reflexions.

The oxazolidinone ring is nearly planar and makes a  $4.5^\circ$  dihedral angle with  
 the adjacent phenyl ring plane. Bond lengths are consistent with a delocalization  
 scheme involving the Ph-N-CO<sub>2</sub> moiety.

(E)-3-[4-[3-NITROPHENYLETHENYL]PHENYL]-5-METHOXYMETHYL-2-OXAZOLIDINONE  
 $C_{19}H_{18}N_2O_5$

F. DURANT, F. LEFEVRE, F. BUFKENS, B. NORBERG and G. EVRARD, 1982. Cryst. Struct. Comm., 11, 1925-1932.

Triclinic,  $P\bar{1}$ ,  $a = 10.729$ ,  $b = 18.283$ ,  $c = 9.982$  Å,  $\alpha = 96.02$ ,  $\beta = 94.47$ ,  $\gamma = 116.59^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.05$  for 3840 reflexions.

The two molecules in the asymmetric unit have similar conformations (Fig. 1). Atom N8 has  $sp^2$  character and indicates electronic delocalization between oxazolidinone and phenyl rings moieties  $Ph-N-CO_2$ .

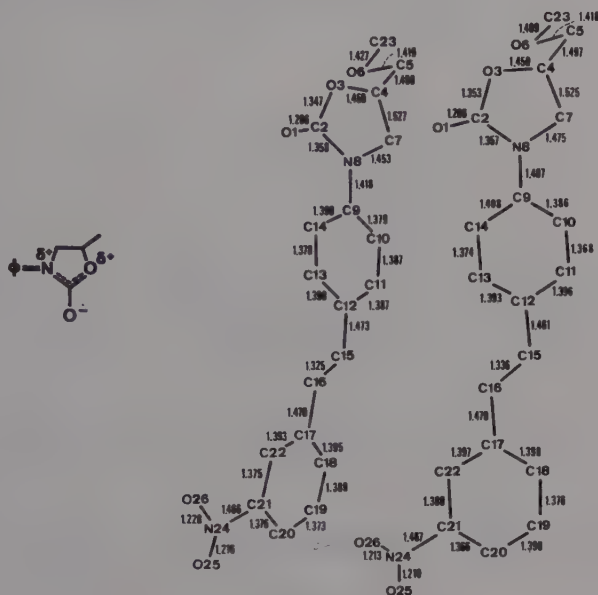
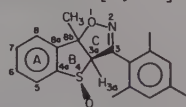


Fig. 1. Bond lengths in the two  $C_{19}H_{18}N_2O_5$  molecules.

anti-4-OXO-8b-METHYL-3-(2,4,6-TRIMETHYLPHENYL)-2,3-DIHYDROBENZO[b]THIOPHENO[2,3-d]-ISOXAZOLINE

$C_{19}H_{19}NO_2S$



A. BENED, R. DURAND, D. PIOCH, P. GENESTE, J.-P. DECLERCQ, G. GERMAIN, J. RAMBAUD, R. ROQUES, C. GUIMON and G.P. GUILLLOUZO, 1982. J. Org. Chem., 47, 2461-2465.

Orthorhombic,  $Pca2_1$ ,  $a = 8.597$ ,  $b = 12.891$ ,  $c = 15.464$  Å,  $Z = 4$ . Cu radiation,  $R = 0.064$  for 1165 reflexions.

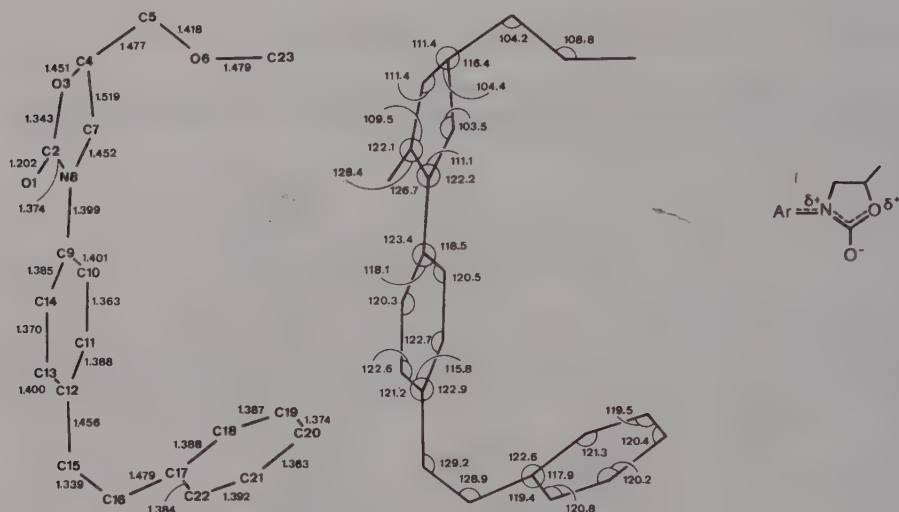
The analysis shows the anti configuration for the molecule.

(Z)-5-(METHOXYMETHYL)-3-[4-(PHENYLETHENYL)PHENYL]-2-OXAZOLIDINONE  
 $C_{19}H_{19}NO_3$

F. DURANT, F. LEFEVRE, B. NORBERG and G. EVRARD, 1982. Cryst. Struct. Comm., 11, 983-990.

Monoclinic,  $P2_1/c$ ,  $a = 11.409$ ,  $b = 12.636$ ,  $c = 11.221$  Å,  $\beta = 90.12^\circ$ ,  $D_m = 1.26$ ,  $Z = 4$ . Cu radiation,  $R = 0.045$  for 1587 reflexions.

The oxazolidinone and phenyl rings are nearly coplanar ( $3.5^\circ$ ). The dimensions are consistent with the delocalization scheme shown.

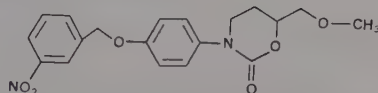


Monoclinic,  $P2_1/c$ ,  $a = 14.097$ ,  $b = 15.309$ ,  $c = 8.679$  Å,  $\beta = 104.48^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.059$  for 1188 reflexions.

The oxazolidinone ring is nearly planar (Fig. 1) and the adjacent phenyl ring is inclined at  $7.4^\circ$  to the oxazolidinone plane. Atom N8 is  $sp^2$  hybridized indicating delocalization in the Ph-N-CO<sub>2</sub> moiety.

3-[4-[(3-NITROPHENYL)METHOXY]PHENYL]-6-METHOXYMETHYL-2-OXAZINONE

$C_{19}H_{20}N_2O_6$



F. DURANT, F. BUFKENS, G. EVRARD and C. LAMOTTE, 1982. Bull. Soc. Chim. Belg., 91, 825-830.

Orthorhombic,  $Pbca$ ,  $a = 39.722$ ,  $b = 16.385$ ,  $c = 10.982$  Å,  $D_m = 1.37$ ,  $Z = 16$ . Cu radiation,  $R = 0.09$  for 2218 reflexions.

There are two independent molecules with similar conformations in the asymmetric unit. The oxazinone ring plane is inclined at  $119-121^\circ$  to the plane of the adjacent phenyl ring. Molecular dimensions are consistent with a resonance scheme involving delocalisation involving the N-CO<sub>2</sub> moiety of the oxazinone ring.

5-BENZYL-10b-HYDROXY-2-ISOPROPYLPYRROLO[3,2-a]PYRROLO[2,1-c]PYRAZINE-3,6-DIONE

$C_{19}H_{24}N_2O_4$

G. LUCENTE, F. PINNEN, G. ZANOTTI, S. CERRINI, F. MAZZA, A.L. SEGRE and W. FEDELI, 1982. J. Chem. Soc. Perkin II, 1169-1174.

Monoclinic,  $P2_1$ ,  $a = 6.794$ ,  $b = 14.379$ ,  $c = 9.260$  Å,  $\beta = 92.75^\circ$ ,  $D_m = 1.26$ ,  $Z = 2$ . Mo radiation,  $R = 0.039$  for 1892 reflexions.

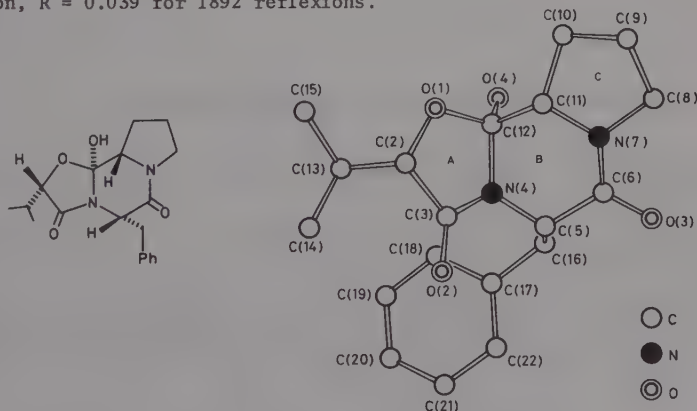


Fig. 1. Molecular skeleton and structure of  $C_{19}H_{24}N_2O_4$ .

This tricyclic peptide cyclol (Fig. 1) is related to the peptide portion of the ergot alkaloids. Ring A adopts an approximate  $C(2)-N(4)-C(12)$  half chair conformation with small torsion angles. Ring B is a half boat with approximate  $C_s$  symmetry through C(6) and C(12) and ring C has a  $C_s$  envelope conformation with C(10) displaced 0.603 Å out of the mean plane of the other four atoms. The amide nitrogen N(4) is significantly pyramidal: the sum of the bond angles round it is  $356.9^\circ$  and it lies 0.143 Å out of the plane of its three substituents. There is an intermolec-

ular hydrogen bond between O(3) and the O(4)H of a molecule translated one unit along *a*, with O(3)...O(4)' 2.772 and O(3)...HO(4)' 1.993 Å.

endo-11-MORPHOLINO-11-SUCCINIMIDO-cis-BICYCLO[8.1.0]UNDECANE

$C_{19}H_{30}N_2O_3$

E. VILSMAIER, C.M. KLEIN, D. DAUSMANN and G. MAAS, 1982. Chem. Ber., 115, 1209-1223.

Orthorhombic,  $P2_12_12_1$ , *a* = 18.900, *b* = 9.952, *c* = 9.693 Å, *Z* = 4. Mo radiation, *R* = 0.042 for 863 reflexions.

The ten-membered ring (Fig. 1) conformation can be described as a boat-chair-boat. Bond lengths and angles have normal values.

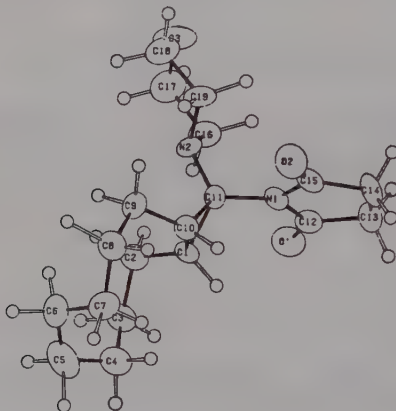


Fig. 1. The structure of  $C_{19}H_{30}N_2O_3$ .

3-[4-[3-CYANOPHENYLETHYNYL]PHENYL]-5-METHOXYMETHYL-2-OXAZOLIDINONE

$C_{20}H_{16}N_2O_3$

F. DURANT, F. LEFÈVRE, F. BUFKENS, G. EVRARD and A. MICHEL, 1982. Bull. Soc. Chim. Belg., 91, 925-929.

Orthorhombic,  $Pbca$ , *a* = 11.431, *b* = 41.411, *c* = 7.262 Å, *Z* = 8. Cu radiation, *R* = 0.04 for 768 reflexions.

The oxazolidinone (Fig. 1) and adjacent phenyl rings are inclined at 24.3° and the O(1)...C(10) distance is 2.91 Å. Electronic delocalization involving the Ph-N-CO<sub>2</sub> moiety is suggested by the molecular dimensions.

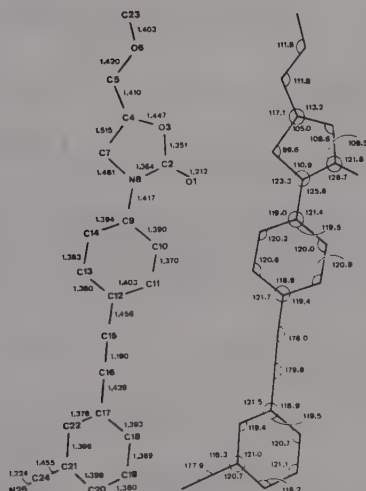


Fig. 1. Molecular dimensions for  $C_{20}H_{16}N_2O_3$ .

5-(1,3-DIMETHYL-5-(2-METHYLPHENYL)-1H-PYRAZOL-4-YL)-3-PHENYL-1,2,4-OXADIAZOLE  
 $C_{20}H_{18}N_4O$

J.B. PRESS, N.H. EUDY, F.M. LOVELL, G.O. MORTON and M.M. SIEGEL, 1982. J. Am. Chem. Soc., 104, 4013-4014.

Monoclinic,  $P2_1/c$ ,  $a = 13.562$ ,  $b = 11.875$ ,  $c = 10.831$  Å,  $\beta = 93.22^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.067$  for 1839 reflexions.

The molecule was shown to contain a 1,2,4-oxadiazole ring and an o-tolyl substituted pyrazole ring (Fig. 1). No details of the molecular geometry were given.

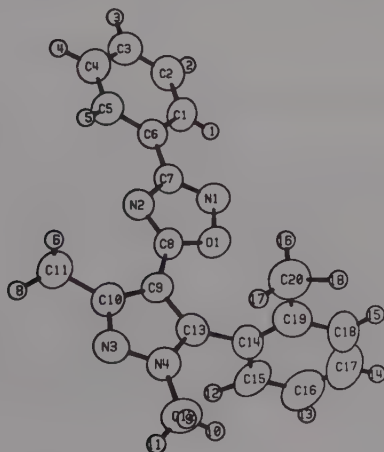


Fig. 1. The  $C_{20}H_{18}N_4O$  molecule.



5-(1-METHYL)-METHOXYMETHYL-3-[4-[(3-CYANOPHENYL)METHOXY]PHENYL]-2-OXAZOLIDINONE  
 $C_{20}H_{20}N_2O_4$

F. DURANT, F. BUFKENS, F. LEFEVRE, C. LAMOTTE and G. EVRARD, 1982. Cryst. Struct. Comm., 11, 1833-1839.

Monoclinic,  $C2/c$ ,  $a = 38.270$ ,  $b = 6.136$ ,  $c = 16.116$  Å,  $\beta = 109.30^\circ$ ,  $D_m = 1.30$ ,  $Z = 8$ . Cu radiation,  $R = 0.099$  for 1811 reflexions.

The oxazolidinone ring is not planar (N8 is 0.023 Å from the plane of the remaining four atoms) and the phenyl ring is inclined at  $21.2^\circ$  to this plane. Atom N8 has  $sp^2$  character indicating delocalization in the Ph-N-CO<sub>2</sub> moiety.

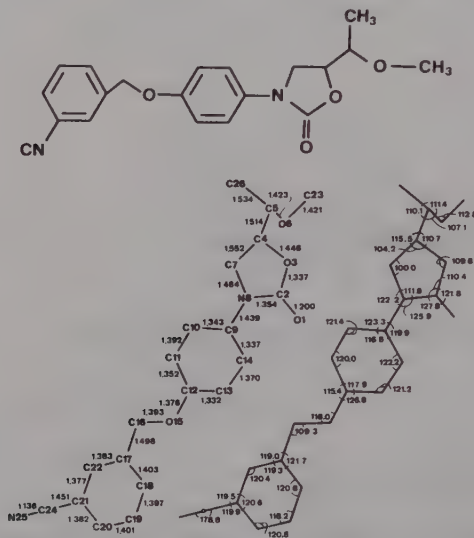


Fig. 1. The  $C_{20}H_{20}N_2O_4$  molecule and dimensions.

2-(2-TOSYLAMINO-5-METHYLPHENYL)BENZOXAZOLE  
 $C_{21}H_{18}N_2O_3S$

Z.A. STARIKOVA, A.E. OBODOVSKAYA and B.M. BOLOTIN, 1982. Zh. Strukt. Khim., 23-1, 128-134 [J. Struct. Chem., 23, 105-110].

Monoclinic,  $P2_1$ ,  $a = 9.117$ ,  $b = 19.216$ ,  $c = 10.822$  Å,  $\beta = 101.84^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.039$  for 2876 reflexions.

The formation of an intramolecular N-H...N hydrogen bond is responsible for the coplanarity of the fused-ring system and the attached phenyl ring (see Fig. 1). The two independent molecules are related by a pseudo-centre of symmetry, forming dimers packed in columns. Average intra- and interdimer distances between planes are 3.3 and 3.5 Å. Bond lengths and angles ( $\sigma = 0.003$ - $0.008$  Å and  $0.2$ - $0.5^\circ$ ) are normal.

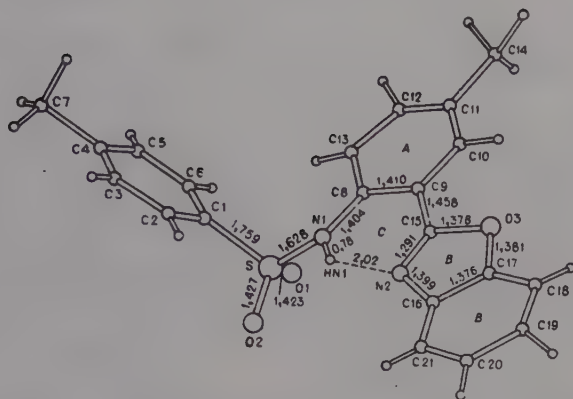


Fig. 1. Structure of  $C_{21}H_{18}N_2O_3S$ , mean bond lengths for the two independent molecules are shown.

ETHYL 10-CHLORO-5,6,7,11b-TETRAHYDRO-7-METHYL-6-OXO-11b-PHENYLISOXAZOLO[2,3-d][1,4]-BENZODIAZEPINE-1-CARBOXYLATE

$C_{21}H_{19}ClN_2O_4$  (I)

[ $\alpha$ -(7-CHLORO-3,4-DIHYDRO-4-METHYL-3-OXO-1(2H)-QUINOXAZOLINYL) BENZYLIDENE]MALONALDEHYDIC ACID ETHYL ESTER

$C_{21}H_{19}ClN_2O_4$  (II)

ETHYL  $\alpha$ -BENZOYL-7-CHLORO-3,4-DIHYDRO-4-METHYL-3-OXO-1(2H)-QUINOXALINEACRYLATE

$C_{21}H_{19}ClN_2O_4$  (III)

J.P. FREEMAN, D.J. DUCHAMP, C.G. CHIDESTER, G. SLOMP, J. SZMUSZKOWICZ and M. RABAN, 1982. J. Am. Chem. Soc., 104, 1380-1386.

I. Monoclinic,  $P2_1/c$ ,  $a = 8.539$ ,  $b = 14.553$ ,  $c = 15.811$  Å,  $\beta = 101.71^\circ$ ,  $Z = 4$ .  
R = 0.089.

II. Monoclinic,  $P2_1/c$ ,  $a = 13.396$ ,  $b = 8.513$ ,  $c = 19.737$  Å,  $\beta = 122.30^\circ$ ,  $Z = 4$ .  
R = 0.094.

III. Monoclinic,  $P2_1/c$ ,  $a = 7.235$ ,  $b = 26.676$ ,  $c = 20.602$  Å,  $\beta = 99.279^\circ$ ,  $Z = 8$ .  
R = 0.105.

The structures of the three isomers are shown in Fig. 1. Compound II and a further rearrangement product, III, could be obtained from I upon treatment with boiling ethanol.

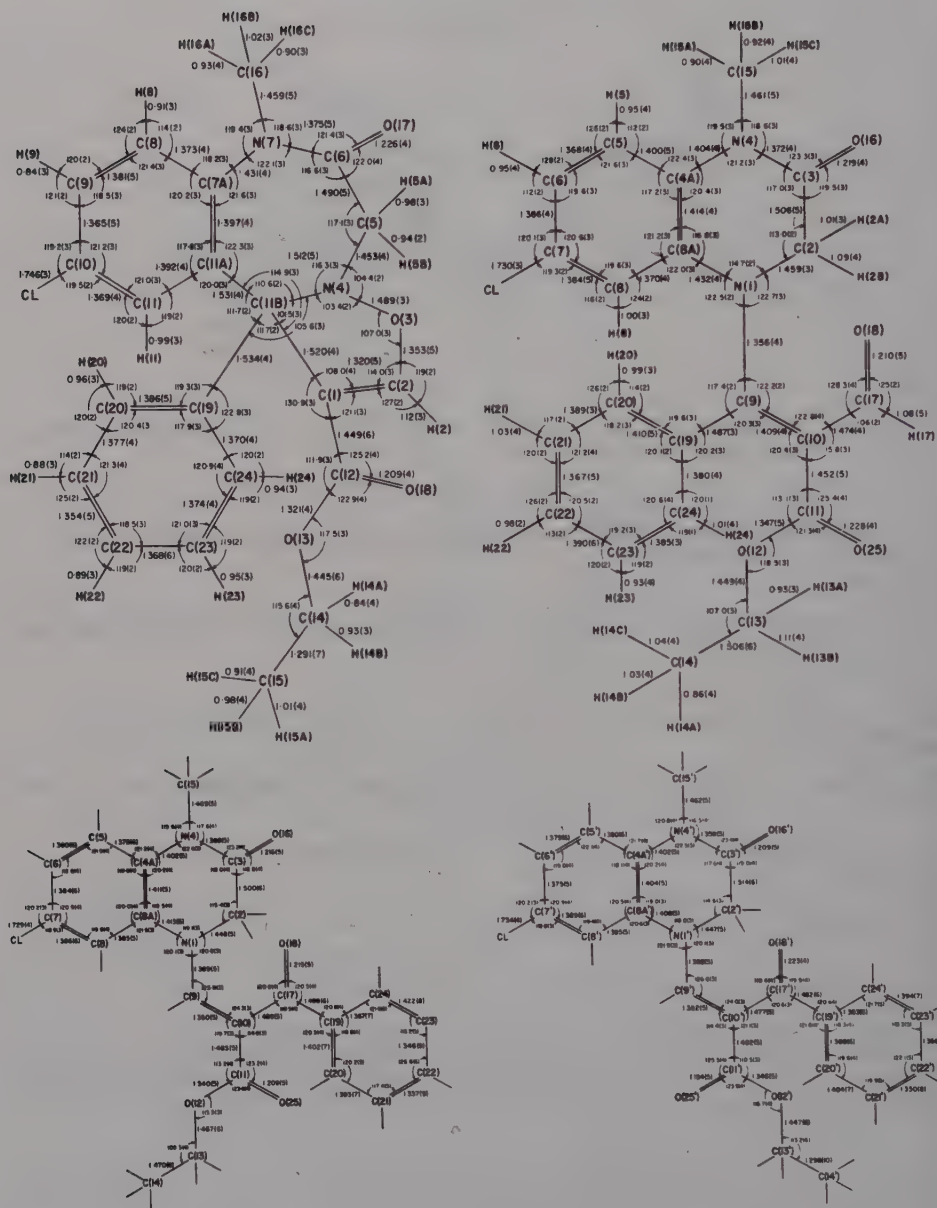


Fig. 1. Bond lengths and angles in  $C_{21}H_{19}ClN_2O_4$  isomers: I (top left), II (top right), and the two independent molecules of III (bottom).



The structure of  $[\text{Na-KRYPTOFIX-221}]^+[\text{Cr}(\text{CO})_5\text{SH}]^-$  consists of an array of the two discrete ionic units at normal van der Waals distances. The sodium ion is hepta-coordinated (Fig. 1) and the bond lengths, polyhedral edge lengths and angles subtended at the  $\text{Na}^+$  ion are provided as supplementary material. In the anion (Fig. 1), the disposition of the ligands about the Cr atom is octahedral where the average  $\text{C}(\text{ax.})-\text{Cr}-\text{C}(\text{eq.})$  angle is  $91.4^\circ$ . The  $\text{Cr}-\text{C}(\text{eq.})$  distances average  $1.904 \text{ \AA}$  while the axial  $\text{Cr}-\text{C}(5)$  bond length is essentially the same at  $1.898 \text{ \AA}$ . The thiol ligand is some  $2.473 \text{ \AA}$  from the metal atom.

t-3-METHYL-r-2,c-4,c-5-TRIPHENYL-1,3-OXAZOLIDINE  
 $\text{C}_{22}\text{H}_{21}\text{NO}$

T. SKARZYŃSKI, 1982. *Acta Cryst.*, **B38**, 3112-3113.

Monoclinic,  $P2_1/a$ ,  $a = 8.880$ ,  $b = 12.015$ ,  $c = 16.557 \text{ \AA}$ ,  $\beta = 90.97^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.044$  for 1585 reflexions.

The methyl group on the N atom (Fig. 1) is on the opposite side of the five-membered ring from the aromatic groups on C(1), C(2) and C(3). The angles between the best planes through the phenyl rings are: I/II  $47.8(2)^\circ$ , I/III  $13.2(2)^\circ$  and II/III  $52.1(2)^\circ$ , where I, II and III represent the phenyl rings on C(1), C(2) and C(3) respectively. The oxazolidine system is in the half-chair conformation, with atoms N and C(1) displaced by  $0.30(2)$  and  $0.36(2) \text{ \AA}$  to opposite sides of the plane through C(2), C(3) and O.

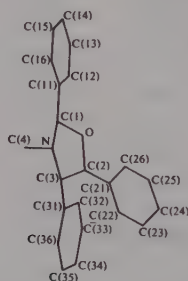


Fig. 1. A view of  $\text{C}_{22}\text{H}_{21}\text{NO}$ .

t-3-METHYL-r-2,c-4,t-5-TRIPHENYL-1,3-OXAZOLIDINE  
 $\text{C}_{22}\text{H}_{21}\text{NO}$

T. SKARZYŃSKI, Z. DEREWENDA, A.M. BRZOZOWSKI and G. MLOSTON, 1982. *Acta Cryst.*, **B38**, 3113-3115.

Monoclinic,  $P2_1/c$ ,  $a = 22.937$ ,  $b = 19.569$ ,  $c = 7.866 \text{ \AA}$ ,  $\beta = 94.74^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.035$  for 2837 reflexions.

There are two independent molecules in the asymmetric unit. Equivalent inter-atomic distances and angles are generally equal within experimental error. Only two angles differ significantly:  $\text{C}(1)-\text{O}-\text{C}(2)$  is  $108.7(1)^\circ$  in molecule A and  $109.7(2)^\circ$  in B (Fig. 1), and  $\text{C}(1)-\text{N}-\text{C}(3)$  is  $102.4(2)^\circ$  in A and  $104.6(2)^\circ$  in B. The angles between planes fitted by least squares to the phenyl rings are as follows: I/II  $38.8(1)^\circ$  in A ( $35.1(1)^\circ$  in B), I/III  $54.8(1)^\circ$  in A ( $57.0(1)^\circ$  in B) and II/III  $47.6(1)^\circ$  in A ( $50.5(1)^\circ$  in B), where I, II and III represent the phenyl rings bonded to C(1), C(2) and C(3) respectively. The geometries of the oxazolidine rings of the two molecules are also somewhat different. The two rings are of the half-chair type. In molecule A atoms N and C(3) are  $0.43(2)$  and  $0.25(2) \text{ \AA}$  from the plane defined by atoms C(1), O, C(2), while in molecule B the equivalent distances are  $0.27(2)$  and  $0.37(2) \text{ \AA}$ . These differences indicate a significant flexibility of the oxazolidine system.

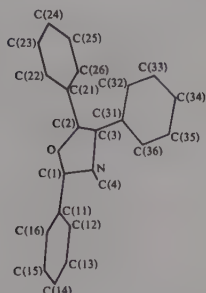
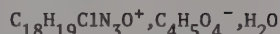
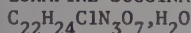


Fig. 1. The skeleton of  $C_{22}H_{21}NO$ .

# LOXAPINE SUCCINATE MONOHYDRATE



J.P. FILLERS and S.W. HAWKINSON, 1982. *Acta Cryst.*, **B38**, 3041-3045.

Triclinic,  $P\bar{1}$ ,  $a = 9.702$ ,  $b = 14.237$ ,  $c = 9.436$  Å,  $\alpha = 92.80$ ,  $\beta = 115.82$ ,  $\gamma = 76.89^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.050$  for 2484 reflexions.

The central seven-membered heterocyclic ring (Fig. 1) is in a boat conformation while the piperazine ring is in the normal chair conformation. The dihedral angle between the planes of the benzene rings is  $121^\circ$ . There are two half-succinate molecules per asymmetric unit, each located on a centre of symmetry. The distal N atom of the piperazine ring hydrogen bonds to one of the succinate O atoms, while the remaining three succinate O atoms hydrogen bond to the water molecule of crystallization.

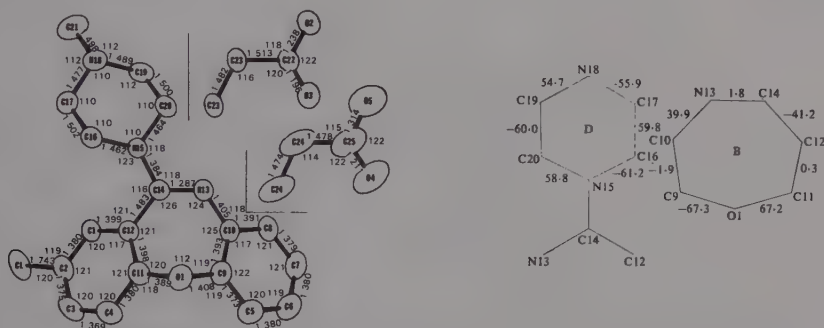
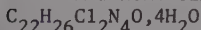


Fig. 1. Dimensions for loxapine succinate monohydrate and torsion angles.

# 3-MORPHOLINOETHYLAMINO-4,6-DIPHENYLPYRIDAZINE DIHYDROCHLORIDE DIHYDRATE



A. MICHEL, R. GUSTIN, G. EVRARD and F. DURANT, 1982. *Bull. Soc. Chim. Belg.*, **91**, 123-129.

Monoclinic,  $P2_1/c$ ,  $a = 17.019$ ,  $b = 7.705$ ,  $c = 22.099$  Å,  $\beta = 118.75^\circ$ ,  $D_m = 1.32$ ,  $Z = 4$ . Cu radiation,  $R = 0.057$  for 3578 reflexions.

Bond lengths and angles are in Fig. 1. Atom N9 has trigonal planar geometry, and a canonical form of the type shown in Fig. 1 is suggested to explain the dimensions of the pyridazine moiety.





Fig. 1. Bond lengths and angles and a possible resonance form of the cation in  $C_{22}H_{26}Cl_2N_4O$ .

2-PHENYL-4-t-BUTYL-5-t-BUTYLIMINO- $\Delta^2$ -1,3,4-OXADIAZOLIUM PICRATE  
 $C_{22}H_{26}N_6O_8$

G.S.D. KING, L. VAN MEERVELT and G. L'ABBE, 1982. Bull. Soc. Chim. Belg., 91, 263-264.

Triclinic,  $P\bar{1}$ ,  $a = 9.812$ ,  $b = 14.775$ ,  $c = 8.618$  Å,  $\alpha = 90.41$ ,  $\beta = 93.28$ ,  $\gamma = 98.17^\circ$ ,  $D_m = 1.36$ ,  $Z = 2$ . Mo radiation,  $R = 0.067$  for 2052 reflexions.

The cation adopts the Z-orientation about the imine function to relieve overcrowding caused by the bulky t-butyl moiety, and the angles (Fig. 1)  $C5-N4-C11$  ( $132^\circ$ ),  $N4-C5-N6$  ( $131^\circ$ ) are larger than  $N3-N4-C11$  ( $119^\circ$ ) and  $O1-C5-N6$  ( $120^\circ$ ). The picrate anion is hydrogen bonded to  $N6$  of the oxadiazolium cation ( $N...O$  2.89 Å).

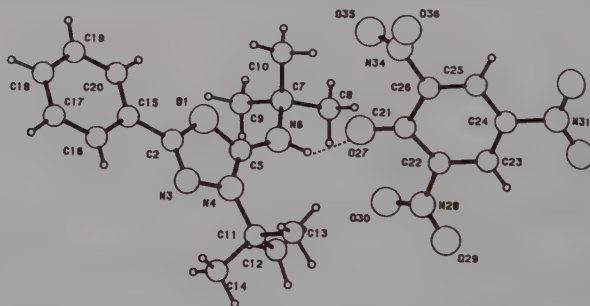


Fig. 1. The  $C_{22}H_{26}N_6O_8$  molecule.

t-3-ETHYL-r-2,c-4,c-5-TRIPHENYL-1,3-OXAZOLIDINE  
 $C_{23}H_{23}NO$

T. SKARŻYŃSKI, 1982. Acta Cryst., B38, 3110-3111.

Orthorhombic,  $Pcab$ ,  $a = 7.641$ ,  $b = 17.324$ ,  $c = 29.042$  Å,  $Z = 8$ . Cu radiation,  $R = 0.044$  for 1781 reflexions.

All three phenyl rings (Fig. 1) are in the trans configuration with respect to the ethyl group bonded to N. The angles between planes fitted by least squares to the phenyl rings are as follows: I/II  $26.5(2)$ , I/III  $26.9(2)$  and II/III  $48.6(3)^\circ$ , where I, II and III represent the phenyl groups on C(1), C(2) and C(3) respectively. The conformation of the oxazolidine ring is of the half-chair type with atoms O and C(1)  $0.35(2)$  and  $0.28(2)$  Å from the plane defined by atoms C(2), C(3) and N. High values of the temperature factors of the atoms, especially those forming the phenyl

rings, result from the diffraction data being collected at a temperature close to the melting point.

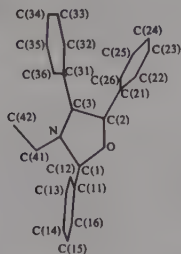
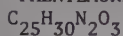


Fig. 1. The  $C_{23}H_{23}NO$  molecule.

(-)-(2R,3S,6'R,1'S)-3-METHYL-N-[6'-(2''-NITRO-1''-PHENYLETHYL)-1-CYCLOHEXENYL]-2-PHENYLMORPHOLINE



S.J. BLARER, W.B. SCHWEIZER and D. SEEBACH, 1982. *Helv. Chim. Acta* **65**, 1637-1654.

Orthorhombic,  $P2_12_12_1$ ,  $a = 6.152$ ,  $b = 15.158$ ,  $c = 23.785$  Å,  $Z = 4$ . Mo radiation,  $R = 0.029$  for 1010 reflexions.

The analysis shows that the molecule has structure and stereochemistry as in Fig. 1.

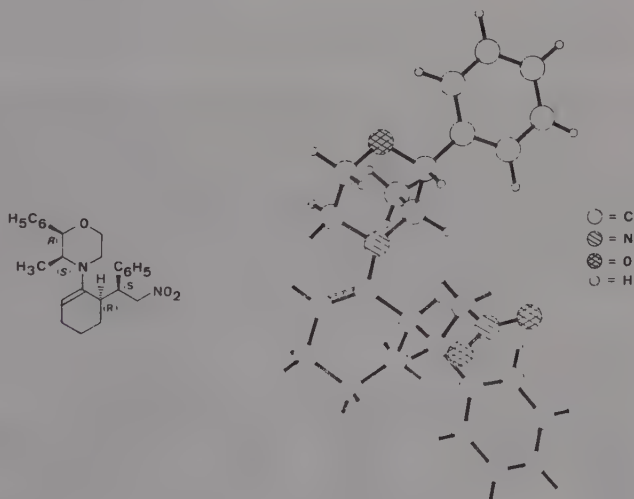
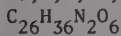


Fig. 1. The  $C_{25}H_{30}N_2O_3$  structure.

4,11,17,24,29,32-HEXAOKA-1,14-DIAZATETRACYCLO[12.12.8.0<sup>5,10</sup>.0<sup>18,23</sup>]TETRATRIACONTA-5,7,9,18,20,22-HEXAENE



N.L. OTT, C.L. BARNES, R.W. TAYLOR and D. VAN DER HELM, 1982. *Acta Cryst.*, **B38**, 2277-2280

Monoclinic,  $P2_1/c$ ,  $a = 11.512$ ,  $b = 8.493$ ,  $c = 27.35$  Å,  $\beta = 112.16^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.066$  for 5087 reflexions (at 138 K).

The molecule (Fig. 1) has the endo-endo conformation. The mean  $C(sp^3)-C(sp^3)$  bond length is 1.511(9) Å, shorter than expected for aliphatic C-C bonds but consistent with values found in many macrocyclic polyethers. The mean aromatic C-C bond length is 1.392(12) Å, close to the expected value. There is no possibility of hydrogen bonding in the crystal.

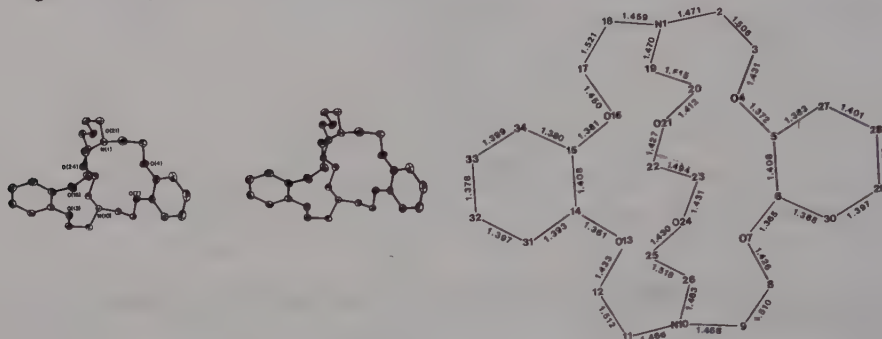


Fig. 1.  $C_{26}H_{36}N_2O_6$ : stereoview and bond distances.

TRIS(MORPHOLINO)SULPHONIUM TETRAPHENYLBORATE  
 $C_{36}H_{44}BN_3O_3S$



C. RØMMING, G.O. NEVSTAD and J. SONGSTAD, 1982. Acta Chem. Scand., A36, 407-415.

Monoclinic,  $P2_1/c$ ,  $a = 9.483$ ,  $b = 19.890$ ,  $c = 17.350$  Å,  $\beta = 90.29^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.069$  for 6188 reflexions (at  $-150^\circ C$ ).

The cation is highly asymmetric (Fig. 1) with two smaller N-S-N angles ( $N(1)-S-N(2)$   $99.0^\circ$ ,  $N(1)-S-N(3)$   $97.3^\circ$ ) and one larger ( $N(2)-S-N(3)$   $117.4^\circ$ ), and two shorter S-N bond lengths (S-N(2) 1.620, S-N(3) 1.636 Å) with one significantly longer (S-N(1) 1.683 Å). The anion (Fig. 1) has slightly distorted tetrahedral geometry and the average B-C bond is 1.645 Å. The phenyl groups are significantly distorted from hexagonal symmetry, the internal angles at the C atoms linked to the central B atom averaging only  $115.6^\circ$ .

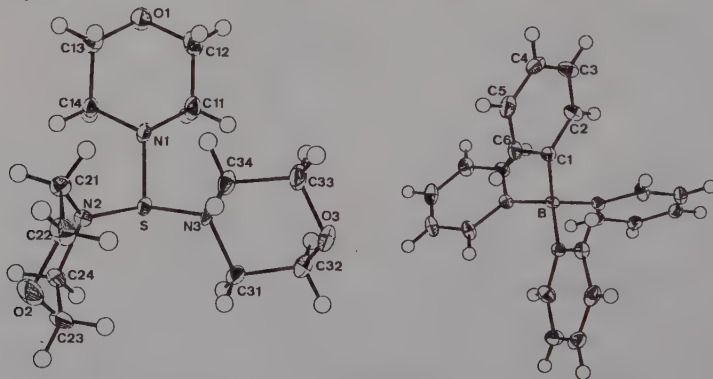
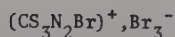
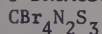


Fig. 1. Molecular structure of the cation (left) and anion (right) in  $C_{36}H_{44}BN_3O_3S$ .

## 5-BROMOSULPHENYL-1,3,2,4-DITHIADIAZOLIUM TRIBROMIDE



G. WOLMERSHÄUSER, C. KRÜGER and Y.-H. TSAY, 1982. Chem. Ber., 115, 1126-1131.

Orthorhombic,  $\text{Pna}2_1$ ,  $a = 7.550$ ,  $b = 12.466$ ,  $c = 10.275$  Å,  $Z = 4$ . Mo radiation,  $R = 0.048$  for 1057 reflexions.

The structure (Fig. 1) is ionic. The cation is planar ( $\pm 0.02$  Å) ( $\text{N}(1)-\text{S}(1)$  1.67(1),  $\text{N}(1)-\text{S}(2)$  1.56(2),  $\text{S}(2)-\text{N}(2)$  1.60(2),  $\text{N}(2)-\text{C}$  1.37(2),  $\text{C}-\text{S}(1)$  1.76(1),  $\text{C}-\text{S}(3)$  1.72(1),  $\text{S}(3)-\text{Br}(4)$  2.208(4) Å); the anion is linear ( $\text{Br}(1)-\text{Br}(2)$  2.721(1),  $\text{Br}(2)-\text{Br}(3)$  2.417(2) Å,  $\text{Br}-\text{Br}-\text{Br}$  178.8(1)°). There are two short interionic contacts:  $\text{Br}(1)-\text{S}(1)$  3.111(4),  $\text{Br}(1)-\text{S}(3)$  3.121(4) Å.

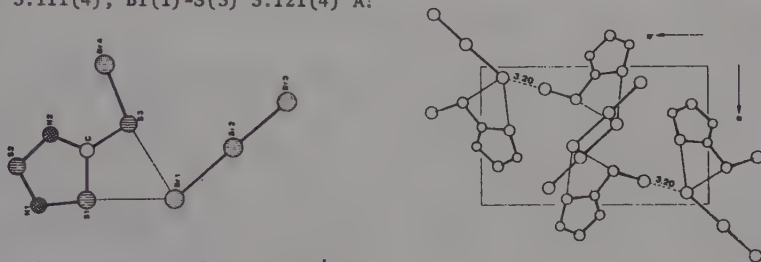


Fig. 1. The structure of  $(\text{CS}_3\text{N}_2\text{Br})^+, \text{Br}_3^-$ .

## 3,5-DIAMINO-1,2,4-DITHIAZOLIUM BROMIDE



G. PEYRONEL, A. PIGNEDOLI and W. MALAVASI, 1982. J. Cryst. Spectrosc. Res., 12, 481-488.

Monoclinic,  $\text{P}2_1/\text{n}$ ,  $a = 9.948$ ,  $b = 12.731$ ,  $c = 5.098$  Å,  $\beta = 98.70^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.036$  for 856 reflexions.

The S-S distance (2.075(2) Å) (Fig. 1), is intermediate between those of the thiouret hydrochloride and hydroiodide (1,2). The C-N (terminal) and C-N (bridging) distances are close to their average value (1.322 Å) within their standard deviations. The  $\nu(\text{NH}_2)$  and  $\delta(\text{NH}_2)$  frequencies of the thiouret hydrohalides are close to those of dithiobiuret while the  $\nu(\text{NH})$  and  $\delta(\text{NH})$  bands are, as expected, absent. The  $\nu(\text{CN})$  and  $\nu(\text{CS})$  bands are shifted to higher and lower frequencies, respectively, while a new  $\nu(\text{SS})$  broad band is observed in the 430-410  $\text{cm}^{-1}$  region.

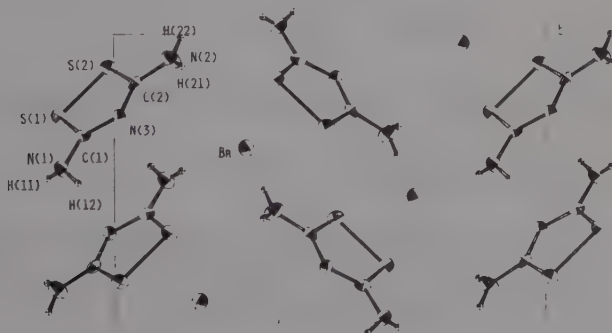


Fig. 1. A projection of  $\text{C}_2\text{H}_4\text{BrN}_3\text{S}_2$ .

1. Structure Reports, 31B, 156.

2. Ibid, 37B, 199.

SODIUM 3-HYDROXY-4-NITRO-6H-1,2,6-THIADIAZINE 1,1-DIOXIDE HYDRATE  
 $C_3H_2N_3NaO_5S \cdot H_2O$  (I)

$Na^+ \cdot C_3H_2N_3O_5S^-, H_2O$

POTASSIUM 3-HYDROXY-4-NITRO-6H-1,2,6-THIADIAZINE 1,1-DIOXIDE HYDRATE  
 $C_3H_2KN_3O_5S \cdot H_2O$  (II)

$K^+ \cdot C_3H_2N_3O_5S^-, H_2O$

C. ESTEBAN-CALDERÓN, M. MARTÍNEZ-RIPOLL and S. GARCÍA-BLANCO, 1982. *Acta Cryst.*, B38, 1124-1128.

I. Monoclinic,  $P2_1/c$ ,  $a = 6.2914$ ,  $b = 14.001$ ,  $c = 8.9204$  Å,  $\beta = 95.382^\circ$ ,  $Z = 4$ .  
 Mo radiation,  $R = 0.041$  for 1945 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 6.0744$ ,  $b = 18.533$ ,  $c = 7.497$  Å,  $\beta = 91.68^\circ$ ,  $Z = 4$ .  
 Mo radiation,  $R = 0.039$  for 2006 reflexions.

Both structures (Fig. 1) contain dimeric thiadiazine molecules formed through two centrosymmetric N-H...O intermolecular hydrogen bonds. In the sodium salt (I) the anion acts as a bidentate ligand for  $Na^+$  ions with two identical  $Na \cdots O$  contacts of 2.40 Å. In the potassium salt (II) the corresponding  $K \cdots O$  contacts are 2.88 and 3.17 Å and so the anion seems to behave as a monodentate ligand. The  $Na^+$  ions are octahedrally coordinated with  $Na \cdots O$  2.34 to 2.49 Å. The  $K^+$  ions are mainly surrounded by seven neighbours with  $K \cdots O$  2.78 to 3.01 Å, forming a laterally capped trigonal prism. Strong electron modifications are found within the thiazine rings of these salts when compared with the free heterocyclic compound.

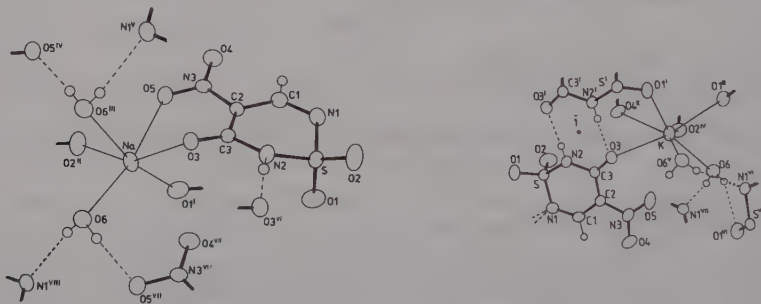


Fig. 1. Perspective drawings of  $Na^+ \cdot C_3H_2N_3O_5S^-, H_2O$  (I) (left) and  $K^+ \cdot C_3H_2N_3O_5S^-, H_2O$  (II) (right).

3-HYDROXY-4-NITRO-6H-1,2,6-THIADIAZINE 1,1-DIOXIDE  
 $C_3H_2N_3O_5S$

C. ESTEBAN-CALDERÓN, M. MARTÍNEZ-RIPOLL and S. GARCÍA-BLANCO, 1982. *Acta Cryst.*, B38, 1340-1342.

Monoclinic,  $P2_1/c$ ,  $a = 12.796$ ,  $b = 5.2427$ ,  $c = 9.7408$  Å,  $\beta = 103.272^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.034$  for 1642 reflexions.

In the molecule (Fig. 1) the thiadiazine ring is in the envelope conformation, the S atom being at the flap. Bond distances and angles indicate electron delocalization within the ring (S-N(1) 1.674, S-N(2) 1.574(1), N(1)-C(1) 1.326, C(1)-C(2) 1.370, C(2)-C(3) 1.442, C(2)-N(3) 1.436, C(3)-N(2) 1.309(2) Å). The molecules are held together in the crystal by hydrogen bonds.

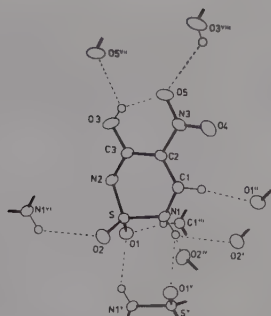


Fig. 1.  $C_3H_3N_3O_5S$ : a perspective drawing of the molecule, broken lines indicate hydrogen bonds.

## 2-AMINO-1,3-THIAZOLE



C. CARANONI and J.P. REBOUL, 1982. *Acta Cryst.*, **B38**, 1255-1259.

Orthorhombic,  $Pbca$ ,  $a = 9.625$ ,  $b = 5.898$ ,  $c = 15.592$  Å,  $D_m = 1.50$ ,  $Z = 8$ . Cu radiation,  $R = 0.026$  for 252 reflexions.

In the molecule (Fig. 1) the exocyclic N atom, slightly out of the thiazole ring plane, is linked by hydrogen bonds to two endocyclic N atoms of two symmetry-related molecules. The crystal stability is also assured by other observed interactions, mainly by C...H contacts ranging from 2.85 to 2.89 Å.

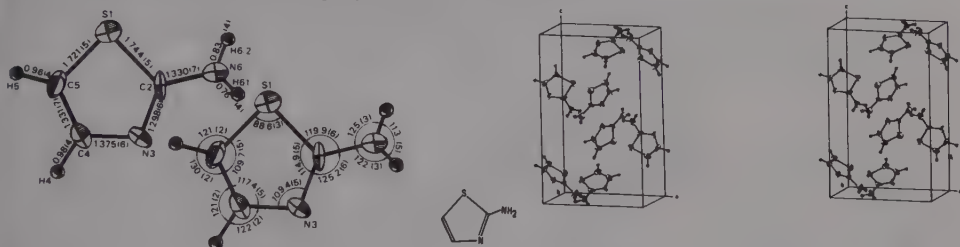
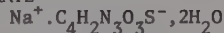
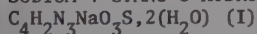
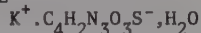
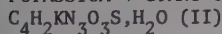


Fig. 1.  $C_3H_4N_2S$ : bond distances and angles and a stereoscopic view of the unit cell.

## SODIUM 4-CYANO-3-HYDROXY-6H-1,2,6-THIADIAZINE 1,1-DIOXIDE DIHYDRATE



## POTASSIUM 4-CYANO-3-HYDROXY-6H-1,2,6-THIADIAZINE 1,1-DIOXIDE HYDRATE



C. ESTEBAN-CALDERÓN, M. MARTÍNEZ-RIPOLL and S. GARCÍA-BLANCO, 1982. *Acta Cryst.*, **B38**, 1128-1133.

I. Triclinic,  $P\bar{1}$ ,  $a = 10.384$ ,  $b = 7.138$ ,  $c = 6.305$  Å,  $\alpha = 108.65$ ,  $\beta = 102.37$ ,  $\gamma = 95.63^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.035$  for 2672 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 9.811$ ,  $b = 7.296$ ,  $c = 5.858$  Å,  $\alpha = 103.06$ ,  $\beta = 94.56$ ,  $\gamma = 100.51^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.042$  for 2835 reflexions.

Both structures (Fig. 1) contain dimeric thiadiazine molecules formed through two centrosymmetric N-H...O intermolecular hydrogen bonds. In the sodium salt (I)



the anion seems to act as a bidentate ligand for the  $\text{Na}^+$  ions whilst in the potassium salt (II) it behaves as a monodentate ligand for the  $\text{K}^+$  ions. The  $\text{Na}^+$  ions are octahedrally coordinated, with  $\text{Na}\cdots\text{O}$  contacts of 2.35 to 2.54 Å, whilst the  $\text{K}^+$  ions are surrounded by seven neighbours with  $\text{K}\cdots\text{O}$  contacts of 2.75 to 2.93 Å. The thiadiazine rings show strong electron modification when compared with the free heterocyclic compound.

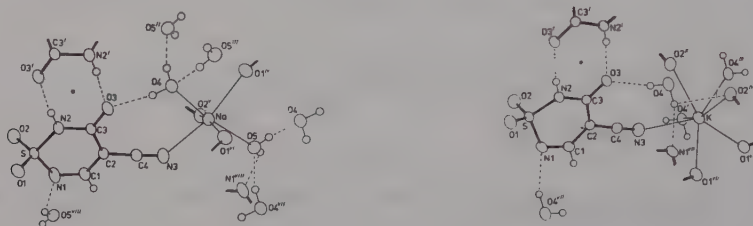


Fig. 1. Perspective drawings of  $\text{Na}^+\cdot\text{C}_4\text{H}_2\text{N}_3\text{O}_3\text{S}^-\cdot 2\text{H}_2\text{O}$  (I) (left) and  $\text{K}^+\cdot\text{C}_4\text{H}_2\text{N}_3\text{O}_3\text{S}^-\cdot \text{H}_2\text{O}$  (right).

# 5-ACYLIMINO-1,2,3-THIADIAZOLE $\text{C}_4\text{H}_5\text{N}_3\text{O}_2\text{S}$

S. BRÜCKNER and L. MALPEZZI, 1982. *Cryst. Struct. Comm.*, **11**, 529-532.

Monoclinic,  $P2_1/c$ ,  $a = 8.426$ ,  $b = 9.475$ ,  $c = 8.539$  Å,  $\beta = 101.2^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.036$  for 1022 reflexions.

The five-membered ring (Fig. 1) is planar and the dimensions are consistent with a  $\pi$ -system extending through the ring and continuing toward C(3). Molecules are linked by  $\text{N-H}\cdots\text{N}$  hydrogen bonds ( $\text{N}(2)\cdots\text{N}(3)$  2.85 Å).

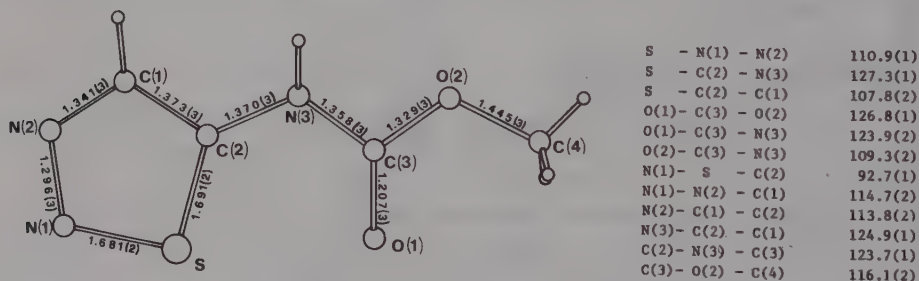


Fig. 1. The  $\text{C}_4\text{H}_5\text{N}_3\text{O}_2\text{S}$  molecule and dimensions.

# 3,5-BIS(METHYLMERCAPTO)-1,2,4-THIADIAZOLE (DIMETHYLPERTHIOCYANATE) $\text{C}_4\text{H}_6\text{N}_2\text{S}_3$

G. GATTOW, G. KIEL and R. GERNER, 1982. *Z. Anorg. Chem.*, **488**, 87-93.

Monoclinic,  $P2_1/c$ ,  $a = 7.661$ ,  $b = 11.675$ ,  $c = 9.549$  Å,  $\beta = 116.7^\circ$ ,  $D_m = 1.551$ ,  $Z = 4$ . Mo radiation,  $R = 0.062$  for 1772 reflexions.

The five-membered ring (Fig. 1) is planar within experimental error. The methylmercapto side chains are nearly co-planar with the ring; the maximum displacement is 0.06 Å (for C(3)). Bond lengths are:  $\text{S}(1)-\text{C}(1)$  1.728(3),  $\text{S}(1)-\text{C}(3)$  1.790(9),  $\text{S}(2)-\text{C}(2)$  1.738(8),  $\text{S}(2)-\text{C}(4)$  1.795(5),  $\text{S}(3)-\text{N}(1)$  1.613(5),  $\text{S}(3)-\text{C}(1)$  1.713(4),  $\text{N}(1)-\text{C}(2)$  1.373(6),  $\text{N}(2)-\text{C}(1)$  1.306(7),  $\text{N}(2)-\text{C}(2)$  1.370(4) Å.

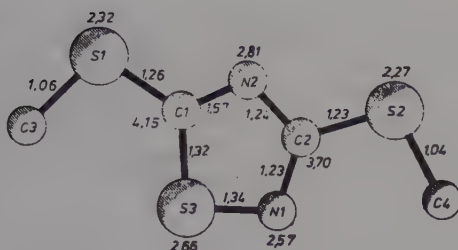


Fig. 1. Bond orders (between atoms) and sums of bond orders (at atoms) in dimethylperthiocyanate.

2',3',4',5'-TETRAHYDROSPIRO(1 $\lambda^4$ ,3 $\lambda^6$ ,5 $\lambda^4$ ,7 $\lambda^4$ ,10-PENTATHIA-2,4,6,8,9,11-HEXAAZABICYCLO-[3.3.3]UNDECA-1(9),2,3,5(11),6,7-HEXAENE-3,1'-THIOPHENE)  
C<sub>4</sub>H<sub>8</sub>N<sub>6</sub>S<sub>5</sub>

H.W. ROESKY, C. PELZ, B. KREBS and G. HENKEL, 1982. Chem. Ber., **115**, 1448-1459.

Monoclinic, P2<sub>1</sub>/c, a = 7.056, b = 13.272, c = 11.660 Å,  $\beta$  = 101.57°, Z = 4. Mo radiation, R = 0.034 for 2053 reflexions.

The molecule as a whole has a 'basket' shape with a N=S(CH<sub>2</sub>)<sub>4</sub>=N group forming the 'handle' (Fig. 1). The S(2),N(2),S(4),N(5),S(3),N(6),S(5),N(3) ring has a saddle conformation while the other two eight-membered rings have a crown arrangement. There is a short S(4)...S(5) intramolecular contact (2.454(1) Å).

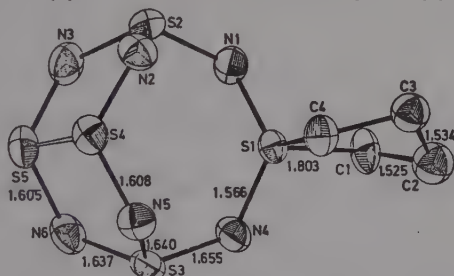


Fig. 1. The structure of C<sub>4</sub>H<sub>8</sub>N<sub>6</sub>S<sub>5</sub> with averaged equivalent bond lengths (Å). E.s.d.'s are 0.002-0.009 Å.

3,5-DIMETHYL-2H-1,2,6-THIADIAZINE-1,1-DIOXIDE  
C<sub>5</sub>H<sub>8</sub>N<sub>2</sub>O<sub>2</sub>S (I)

2,5-DIMETHYL-1,2,6-THIADIAZINE-1,1-DIOXIDE  
C<sub>5</sub>H<sub>8</sub>N<sub>2</sub>O<sub>2</sub>S (II)

J. ELGUERO, C. OCHOA, M. STUD, C. ESTEBAN-CALDERON, M. MARTINEZ-RIPOLL, J.-P. FAYET and M.-C. VERTUT, 1982. J. Org. Chem., **47**, 536-544.

I. Monoclinic, P2<sub>1</sub>/c, a = 9.390, b = 10.452, c = 7.3152 Å,  $\beta$  = 94.64°, Z = 4. Mo radiation, R = 0.033 for 1332 reflexions.

II. Monoclinic, P2<sub>1</sub>/n, a = 12.699, b = 7.227, c = 7.923 Å,  $\beta$  = 92.42°, Z = 4. Mo radiation, R = 0.041 for 1036 reflexions.

The structures of these two derivatives were determined in a comparative study of physicochemical properties of a series of 2H-1,2,6-thiadiazine-1,1-dioxide compounds and their related pyrazole derivatives. The molecular dimensions of the two molecules (Fig. 1) are very similar, with the main differences in the N-S-N region, particularly in the S-N2 distances, 1.672(2) in I, 1.640(3) Å in II, presumably resulting from the methyl substitution at N2 in I. In both compounds, the six-membered rings have envelope shapes, with the S atoms forming the flaps. Bond lengths and bond orders, from the CNDO/2 method, are compared; the N2-C3=C4-C5=N6 system is clearly conjugated.

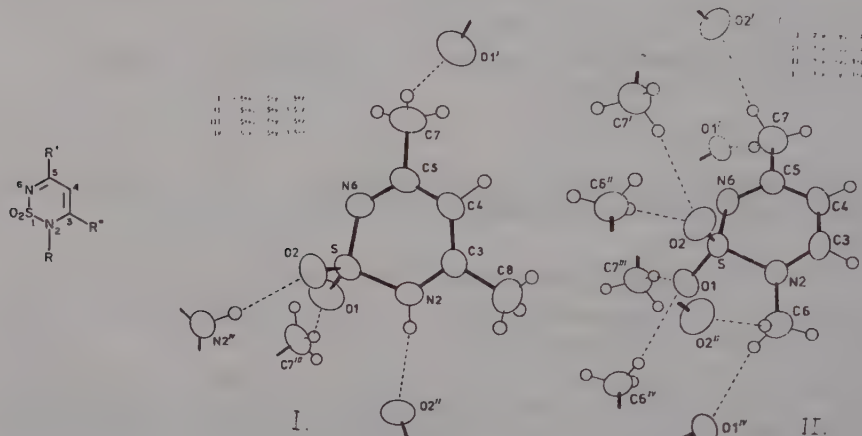


Fig. 1. Thiadiazine ring formula and views of I and II (with intermolecular contacts).

7-AMINO-2,4-DIMETHYL-2H,4H-1,2,3-TRIAZOLO[4,5-c][1,2,6]THIADIAZINE 5,5-DIOXIDE  
 $C_5H_8N_6O_2S$

C. ESTEBAN-CALDERÓN, M. MARTÍNEZ-RIPOLL and S. GARCÍA-BLANCO, 1982. Acta Cryst., B38, 2296-2298.

Triclinic,  $P\bar{1}$ ,  $a = 7.430$ ,  $b = 8.639$ ,  $c = 7.357$  Å,  $\alpha = 105.24$ ,  $\beta = 103.12$ ,  $\gamma = 76.55^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.046$  for 1402 reflexions.

In the molecule (Fig. 1) the thiadiazine ring is in the expected envelope conformation. Considerable electron delocalization is indicated by the bond distances in the molecular rings. The crystal packing is mainly due to N-H...O and N-H...N hydrogen bonds.

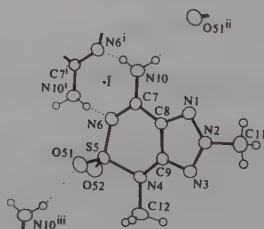
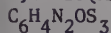


Fig. 1.  $C_5H_8N_6O_2S$ : a perspective drawing showing the molecule; dashed lines represent hydrogen bonds.

## 3,4-BIS(METHYLTHIO)-1,2,5-THIADIAZOLE 1-OXIDE



J.S. AMATO, S. KARADY, R.A. REAMER, H.B. SCHLEGEL, J.P. SPRINGER and L.M. WEINSTOCK, 1982. *J. Am. Chem. Soc.*, **104**, 1375-1380.

Monoclinic,  $P2_1/c$ ,  $a = 4.174$ ,  $b = 13.230$ ,  $c = 14.814$  Å,  $\beta = 93.41^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.068$  for 1020 reflexions.

The molecule (Fig. 1) contains a slightly nonplanar and nonaromatic ring with a pyramidal sulfoxide group. Mean bond lengths are: S-N 1.712(5), N-C 1.280(7), C-C 1.511(8), S-Me 1.782(6), and S-O 1.441(6) Å.

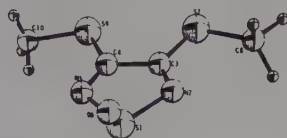
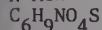


Fig. 1. The  $\text{C}_6\text{H}_4\text{N}_2\text{OS}_3$  molecule.

## N-ACETYL-4-CARBOXY-1,3-THIAZOLIDINE 1-OXIDE



B. TINANT, J.P. DECLERCQ, G. GERMAIN and M. VAN MEERSSCHE, 1982. *Cryst. Struct. Comm.*, **11**, 523-527.

Orthorhombic,  $P2_12_12_1$ ,  $a = 11.079$ ,  $b = 10.231$ ,  $c = 7.354$  Å,  $Z = 4$ . Mo radiation,  $R = 0.030$  for 661 reflexions.

The five-membered ring (Fig. 1) has a conformation closer to a half-chair with the  $C_2$  axis passing through C(4) than to an envelope with the sulphur at the flap. The S-O bond (1.501(3) Å) is pseudo-axial. Molecules are linked by O-H...O hydrogen bonds (2.586 Å) between carboxyl hydroxyl and sulfoxide oxygen.

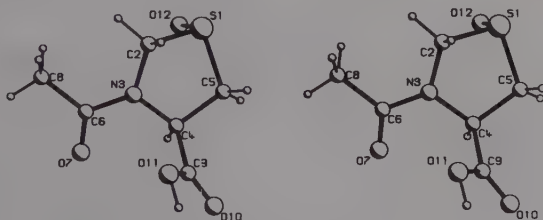
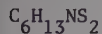


Fig. 1. A stereoview of the  $\text{C}_6\text{H}_9\text{NO}_4\text{S}$  molecule.

## DIHYDRO-2,4,6-TRIMETHYL-4H-1,3,5-DITHIAZINE (THIALDINE)



C.S. DAY, T.J. HANSEN and L.K. KEEFER, 1982. *J. Heterocyclic Chem.*, **19**, 1301-1304.

Monoclinic,  $P2_1/n$ ,  $a = 8.499$ ,  $b = 15.656$ ,  $c = 13.975$  Å,  $\beta = 102.63^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.037$  for 1999 reflexions.

The analysis establishes the all-cis configuration for thialdine (Fig. 1). Both independent molecules have chair conformations with equatorial methyl groups. The S-C distances are in the range 1.803-1.850(4) Å, with N-C 1.428-1.444(4) Å.

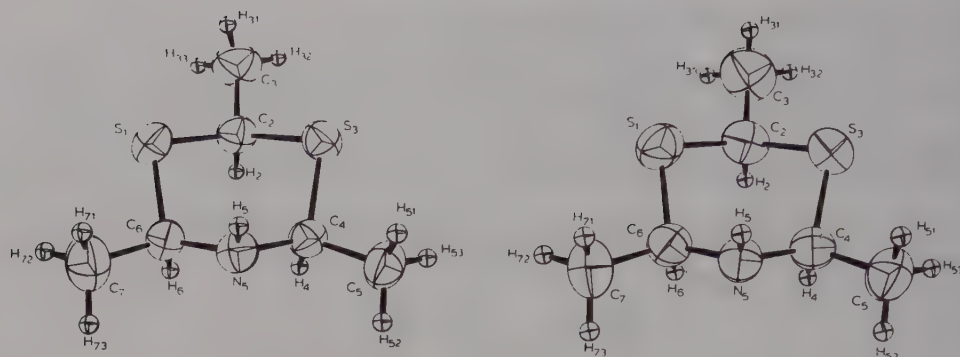


Fig. 1. The two thialdine molecules.

SODIUM SACCHARINATE HYDRATE  
 $C_7H_4NNaO_3S \cdot 0.67(H_2O)$  (I)

MAGNESIUM DISACCHARINATE HEPTAHYDRATE  
 $C_{14}H_{18}MgN_2O_6S_2 \cdot 7H_2O$  (II)

G. JOVANOVSKI and B. KAMENAR, 1982. Cryst. Struct. Comm., **11**, 247-255.

I. Triclinic,  $P\bar{1}$ ,  $a = 15.237$ ,  $b = 11.640$ ,  $c = 7.346$  Å,  $\alpha = 96.31$ ,  $\beta = 87.64$ ,  $\gamma = 107.55^\circ$ ,  $D_m = 1.70$ ,  $Z = 6$ . Mo radiation,  $R = 0.039$  for 5365 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 14.305$ ,  $b = 11.288$ ,  $c = 7.017$  Å,  $\alpha = 97.02$ ,  $\beta = 78.11$ ,  $\gamma = 100.13^\circ$ ,  $D_m = 1.50$ ,  $Z = 2$ . Mo radiation,  $R = 0.038$  for 4769 reflexions.

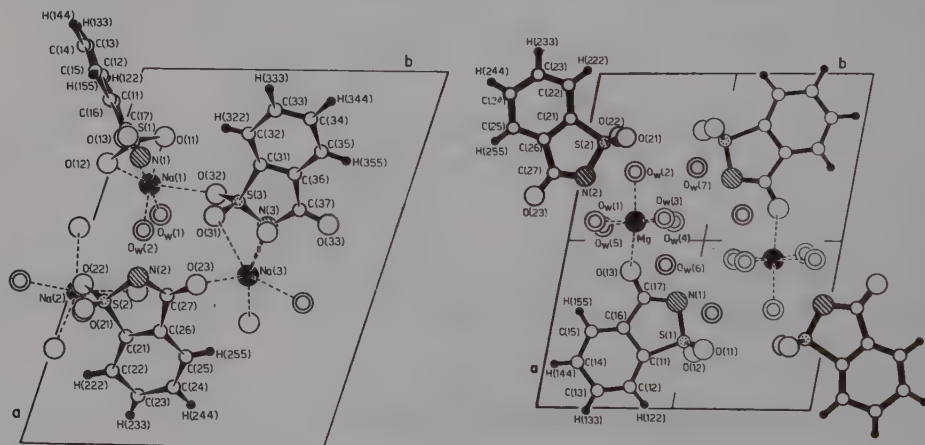


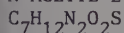
Fig. 1. Views of the saccharinate derivatives.

Both structures (Fig. 1) contain discrete cations, saccharinate anions and water molecules. In (I), two crystallographically independent  $Na^+$  ions have coordination number six, being surrounded by oxygen atoms from water molecules, CO and  $SO_2$  groups from saccharinates, while the third crystallographically independent  $Na^+$  ion has also coordination number six but realized by five oxygen atoms from the same groups and by one saccharinate nitrogen atom. Coordination polyhedra are irregular. Within coordination polyhedra the sodium-to-oxygen distances vary from 2.304 to 2.796 Å, while the sodium-to-nitrogen distance amounts to 2.731 Å. In (II),  $Mg^{2+}$  is octahedrally surrounded by five oxygen atoms from water molecules and one

oxygen atom from the CO group of a neighbouring saccharinate ion. Magnesium-to-oxygen distances range from 2.012 to 2.113 Å, the angles in the octahedron from 86.2 to 96.9°. In both structures the oxygen and nitrogen atoms from saccharinate ions participate in hydrogen bonding with water molecules. Saccharinate ions are planar within the experimental errors. Bond lengths and angles in these ions are within expected values and not very different from those found in saccharin itself (1).

1. Structure Reports, 34B, 207.

N-ACETYL-L-THIAZOLIDINE-4-METHYLCARBOXAMIDE



B. TINANT, J.-P. DECLERCQ, G. GERMAIN and M. VAN MEERSSCHE, 1982. Bull. Soc. Chim. Belg., 91, 57-60.

Hexagonal,  $P6_5$ ,  $a = 8.785$ ,  $c = 20.773$  Å,  $Z = 6$ . Mo radiation,  $R = 0.042$  for 621 reflexions.

The five-membered ring (Fig. 1) has a half-chair conformation. The C-S distances are 1.806 and 1.813(7) Å.

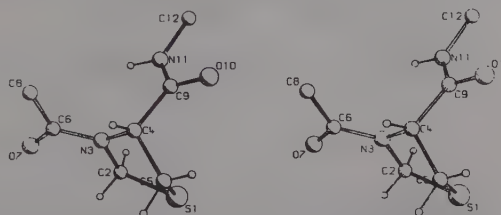
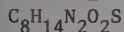


Fig. 1. A stereoview of  $C_7H_{12}N_2O_2S$ .

4-ACETYL-1,4-PERHYDROTHIAZINE-3-METHYLCARBOXAMIDE



B. TINANT, J.P. DECLERCQ, G. GERMAIN and M. VAN MEERSSCHE, 1982. Bull. Soc. Chim. Belg., 91, 61-65.

Orthorhombic,  $Pcab$ ,  $a = 12.797$ ,  $b = 9.923$ ,  $c = 15.533$  Å,  $Z = 8$ . Cu radiation,  $R = 0.073$  for 1186 reflexions.

The six-membered ring is in chair conformation with axial substituents. The C-S distances are 1.804 and 1.806(4) Å, and C-S-C is 96.3(2)°.

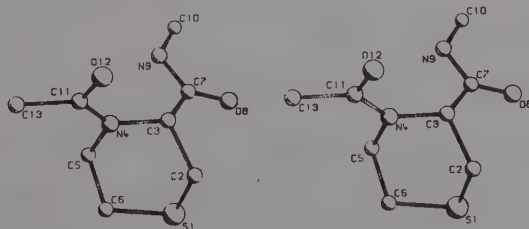
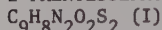


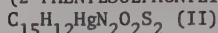
Fig. 1. A stereoview of  $C_8H_{14}N_2O_2S$ .



## 2-PHENYLSULPHONYLIMINO-1,2-DIHYDROTHIAZOLE



## (2-PHENYLSULPHONYLIMINO-1,2-DIHYDROTHIAZOLINYL-N)PHENYLMERCURY



L.G. KUZ'MINA, Yu.T. STRUCHKOV, D.N. KRAVTSOV and L.S. GOLOVCHENKO, 1982. Zh. Struk. Khim., 23-1, 102-110 [J. Struct. Chem., 23, 85-92].

I. Monoclinic,  $P2_1/n$ ,  $a = 5.510$ ,  $b = 8.766$ ,  $c = 20.681 \text{ \AA}$ ,  $\beta = 96.23^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.026$  for 1931 reflexions (at  $-120^\circ\text{C}$ ).

II. Monoclinic,  $P2_1/c$ ,  $a = 15.516$ ,  $b = 10.534$ ,  $c = 22.877 \text{ \AA}$ ,  $\beta = 105.294^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.052$  for 8624 reflexions (at  $-120^\circ\text{C}$ ).

The two structures are shown in Fig. 1. In each case the molecules exist in the imino form, the H atom in I and the HgPh substituent in II being joined to the heterocyclic nitrogen atom N(1). The Hg coordination is linear in II, C-Hg-N  $176.6(4)$  and  $170.3(4)^\circ$  for the two independent molecules.

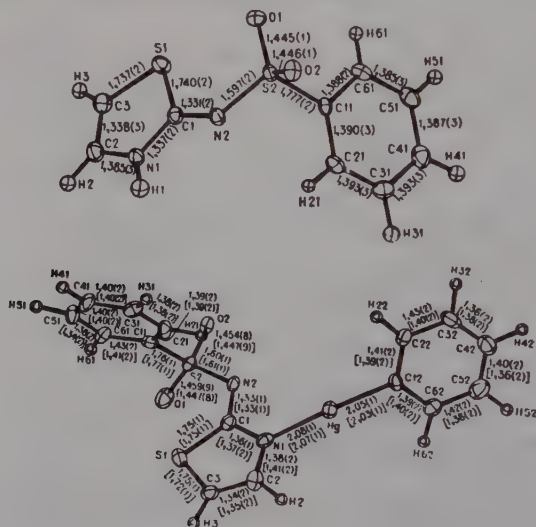
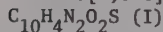
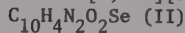


Fig. 1. Bond lengths in  $\text{C}_9\text{H}_8\text{N}_2\text{O}_2\text{S}_2$  (top) and  $\text{C}_{15}\text{H}_{12}\text{HgN}_2\text{O}_2\text{S}_2$  (bottom).

## NAPHTHO[2,3-c][1,2,5]THIADIAZOL-4,9-DIONE



## NAPHTHO[2,3-c][1,2,5]SELENADIAZOL-4,9-DIONE



A. GIEREN and V. LAMM, 1982. Acta Cryst., B38, 2605-2611.

I. Monoclinic,  $P2_1/c$ ,  $a = 16.072$ ,  $b = 8.295$ ,  $c = 13.740 \text{ \AA}$ ,  $\beta = 107.38^\circ$ ,  $D_m = 1.62$ ,  $Z = 8$ . Mo radiation,  $R = 0.048$  for 1868 reflexions.

II. Monoclinic,  $P2_1$ ,  $a = 3.885$ ,  $b = 7.319$ ,  $c = 15.156 \text{ \AA}$ ,  $\beta = 92.14^\circ$ ,  $D_m = 2.01$ ,  $Z = 2$ . Mo radiation,  $R = 0.042$  for 811 reflexions.

Both molecules (Fig. 1) show a significant  $\pi$  interaction of the chalcogen diimide moiety with the  $\pi$  system of the condensed 1,4-naphthoquinone system. The S-N distances are  $1.619(2) \text{ \AA}$  and the Se-N distances  $1.800(6) \text{ \AA}$  on average. The  $\pi$ -bond

order of the S-N bonds is somewhat increased in comparison with the Se-N bonds, and in correlation the  $\pi$  interaction of the chalcogen diimide fragment in I with the substituting 1,4-naphthoquinone system is diminished. The  $\pi$ -bond orders of the chalcogen-nitrogen bonds are also correlated with the length of the short S...O and Se...O contact distances (S...O 3.095(4), 3.146(4), Se...O 2.945(6) Å) occurring in the crystal structure.

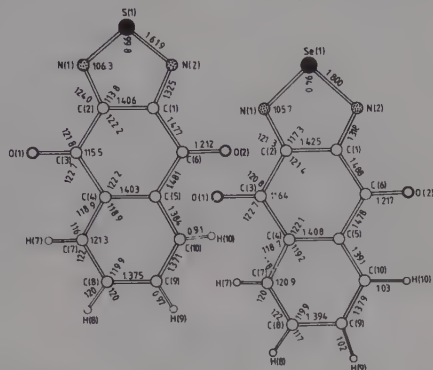


Fig. 1. Bond lengths and angles in  $C_{10}H_4N_2O_2S$  (I) and  $C_{10}H_4N_2O_2Se$  (II).

Z-2-(1-BROMOETHYLIDINE)-2(H)-1,4-BENZOTHAZIN-3(4H)-ONE  
 $C_{10}H_8BrNOS$

R.B. BATES, L.M. DUGUAY, R.E. KLENCK, G.R. KRIEK, M.S. TEMPESTA and A.D. BREWER, 1982. *J. Heterocyclic Chem.*, **19**, 927-928.

Triclinic,  $P\bar{1}$ ,  $a = 4.078$ ,  $b = 10.81$ ,  $c = 12.01$  Å,  $\alpha = 99.9$ ,  $\beta = 96.2$ ,  $\gamma = 94.5^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.093$  for 600 reflexions.

This product of a bromination reaction was shown to have the structure given in Fig. 1. The benzene, amide and alkene groupings are each essentially planar with angles of  $9^\circ$  between benzene and amide and  $22^\circ$  between amide and alkene. The two molecules in a unit cell are held together by two hydrogen bonds across a centre of symmetry; the N-O distance in each hydrogen bond is 2.92(4) Å.



I. Monoclinic,  $P2_1/c$ ,  $a = 9.260$ ,  $b = 11.303$ ,  $c = 13.316$  Å,  $\beta = 128.15^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.068$  for 1217 reflexions.

II. Monoclinic,  $P2_1$ ,  $a = 10.858$ ,  $b = 7.236$ ,  $c = 6.207$  Å,  $\beta = 94.50^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.070$  for 688 reflexions.

III. Triclinic,  $P\bar{1}$ ,  $a = 15.739$ ,  $b = 7.190$ ,  $c = 6.301$  Å,  $\alpha = 70.15$ ,  $\beta = 122.45$ ,  $\gamma = 117.45^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.066$  for 1540 reflexions.

In agreement with NMR results the conformations found are "amino" for I (Fig. 1) and "imino" for II and III (Fig. 1). A short S...O intramolecular distance is found in each compound - possibly constituting a weak bond. The thiazoline rings have the half-chair conformation with C(2) 0.28, 0.49 and 0.18 Å respectively from the C(1),S,N(2),C(3) plane in each molecule. This plane and those of the amide group and benzene ring are almost coplanar in II and III. In I N(1)-H...N(2) hydrogen bonds (2.953(5) Å) link molecules into dimers across a centre of symmetry. In II and III molecules are linked into chains along  $c$  by N(2)-H...O hydrogen bonds of length 2.780(6) and 2.776(4) Å.

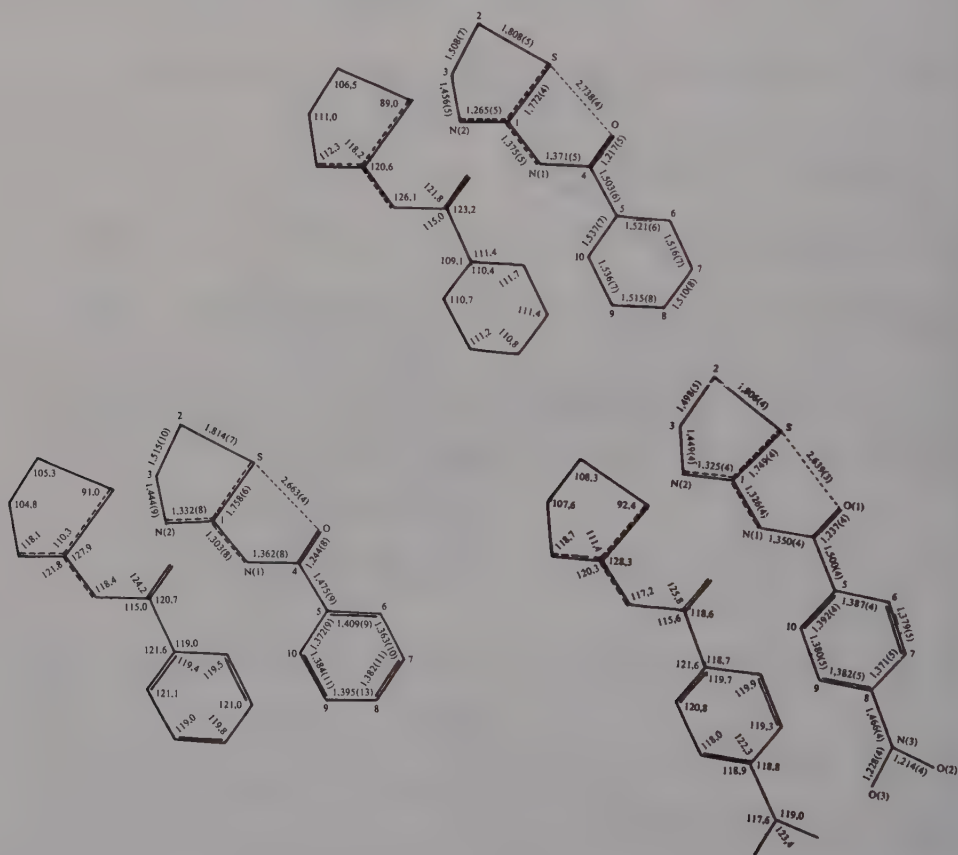


Fig. 1. Bond lengths and angles in 2-(cyclohexanecarbonyl)amino- $\Delta^2$ -1,3-thiazoline (top), in 2-benzoylimino-1,3-thiazolidine (lower left) and in 2-(p-nitrobenzoyl)imino-1,3-thiazolidine (lower right).

2-DIISOPROPYLAMINO-4-METHYL-3,4-DIAZA-1,6,6a-TRITHIAPENTALENYLIUM-5-THIOLATE  
 $C_{10}H_{17}N_3S_4$

L.K. HANSEN, 1982. Acta Chem. Scand., **A36**, 445-449.

Triclinic,  $P\bar{1}$ ,  $a = 5.946$ ,  $b = 9.021$ ,  $c = 13.900$  Å,  $\alpha = 91.06$ ,  $\beta = 99.81$ ,  $\gamma = 98.02^\circ$ ,  $D_m = 1.39$ ,  $Z = 2$ . Mo radiation,  $R = 0.040$  for 1948 reflexions.

The two five-membered rings of the central ring system (Fig. 1) deviate a little from planarity, and the angle between the least squares planes of the rings is  $9.9^\circ$ . Important bond lengths are: S(1)-S(6a) 2.554, S(6a)-S(6) 2.190, S(1)-C(2) 1.693, S(6a)-C(3a) 1.755, S(6)-C(5) 1.706, C(2)-N(3) 1.378, N(3)-C(3a) 1.297, C(3a)-N(4) 1.389, and N(4)-C(5) 1.378 Å. The angle S(1)-S(6a)-S(6) is  $172.99^\circ$ . The bond lengths have been corrected for libration. The S(1)...H(13) and S(7)...H(82) intramolecular contacts are respectively 2.41 and 2.48 Å and there is also a short (3.48 Å) S(6a)...S(6a) intermolecular contact across a symmetry centre.

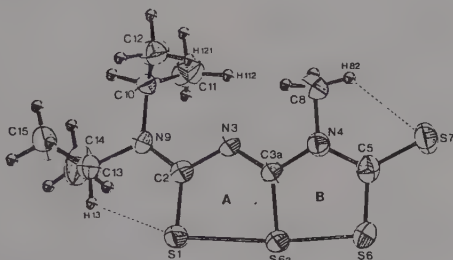


Fig. 1. Molecular structure of  $C_{10}H_{17}N_3S_4$ .

1,1,5,5-TETRAETHYL-3,7-BIS(TRIFLUOROMETHYLSULFONYLIMINO)-1λ<sup>6</sup>,3λ<sup>4</sup>,5λ<sup>6</sup>,7λ<sup>4</sup>,2,4,6,8-TETRAITHIATETRAZOCINE



A. GIEREN, H.W. ROESKY and L. SCHONFELDER, 1982. Z. Anorg. Chem., **493**, 158-170.

Monoclinic,  $P2_1/n$ ,  $a = 11.682$ ,  $b = 7.951$ ,  $c = 12.072$  Å,  $\beta = 96.52^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.039$  for 1729 reflexions.

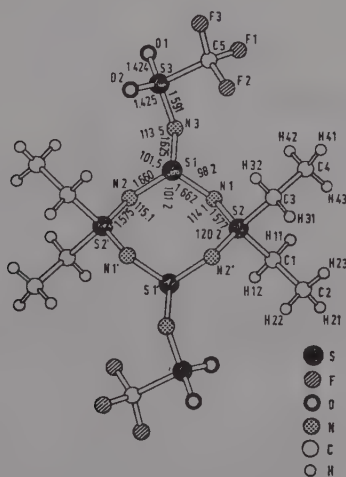


Fig. 1. The structure of  $C_{10}H_{20}F_6N_6O_4S_6$  with bond lengths (Å) and angles ( $^\circ$ ).

The molecule (Fig. 1) has crystallographic  $C_i$ -symmetry. The  $S_4N_4$  ring adopts a chair conformation with almost exact  $C_{2h}$ -symmetry; the four ring N atoms, S(2) and S(2') are coplanar with S(1) and S(1') alternatively above and below this plane by 1.05 Å. The trifluoromethylsulfonylimino substituents occupy equatorial positions. The S-N bond lengths in the pyramidal  $S^{IV}N_3$  moieties are 1.661(3) Å for the endocyclic bonds and 1.625(3) Å for the exocyclic ones. The S-N bonds of the  $S^{VI}N_2$  moieties are significantly shorter at 1.576(3) Å.

3-(2-PYRIDYL)-2-THIOCARBONYL-PYRIDO[3,2-d]THIAZOLIDINE

$C_{11}H_7N_3S_2$

L. DUPONT, O. DIDEBERG and J.C. JAMOULLE, 1982. Cryst. Struct. Comm., 11, 1379-1383.

Monoclinic,  $P2_1/c$ ,  $a = 9.966$ ,  $b = 12.932$ ,  $c = 8.772$  Å,  $\beta = 107.95^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.089$  for 1725 reflexions.

The analysis confirms the proposed formulation of the molecule (Fig. 1). The dihedral angle between the pyridothiazolidine and pyridine ring planes is  $67.3(9)^\circ$ . There is no hydrogen bonding in the structure.

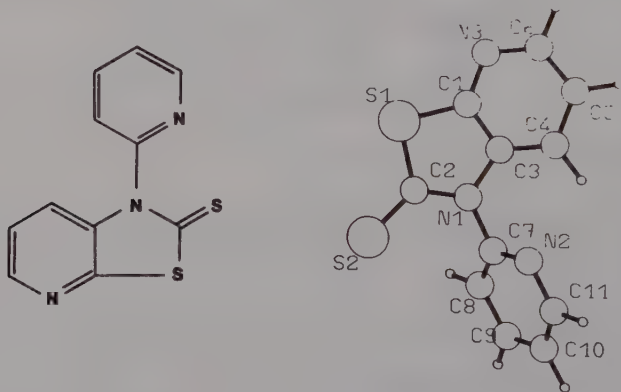


Fig. 1. A view of  $C_{11}H_7N_3S_2$ .

2-ACETYL-3-METHYL-4H-1,4-BENZOTHAZINE

$C_{11}H_{11}NOS$

G. FERGUSON and B. RUHL, 1982. Cryst. Struct. Comm., 11, 1033-1038.

Monoclinic,  $P2_1/c$ ,  $a = 7.392$ ,  $b = 7.683$ ,  $c = 17.561$  Å,  $\beta = 98.15^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.056$  for 856 reflexions.

The dimensions are in accord with the resonance hybrid formulation shown in Fig. 1, with N-C(2) 1.356(6), C(2)-C(3) 1.371(6), C(3)-C(4) 1.453(6) and C(4)-O 1.224(5) Å. Molecules are linked by N-H...O hydrogen bonds (N...O 2.888 Å).

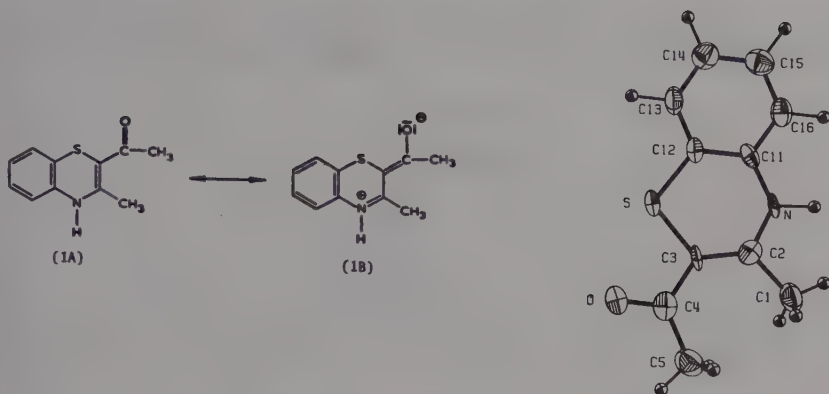


Fig. 1.  $C_{11}H_{11}NOS$ : resonance forms and numbering scheme.

2,2,6,6,9-PENTAMETHYL-4,1,7,9-THIATRIAZATRICYCLO[5.3.0.0<sup>3,5</sup>]DEC-3(5)-ENE-4,8,10-TRIONE

$C_{11}H_{15}N_3O_3S$

W. ANDO, Y. HANYU and T. TAKATA, 1982. J. Am. Chem. Soc., 104, 4981-4982.

Monoclinic,  $P2_1/n$ ,  $a = 15.208$ ,  $b = 6.083$ ,  $c = 16.139$  Å,  $\beta = 117.06^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.058$  for 1691 reflexions.

The most interesting feature of this structure is the geometry of the three-membered  $SC_2$  ring (see Fig. 1) in which all three bonds, as well as the O-S bond, are short. This increase in  $\pi$ -bond character in the  $O=SC_2$  moiety may be attributed to the combined effects of ring fusion and alkyl substitution.

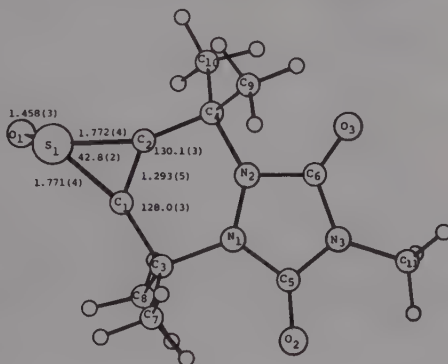


Fig. 1. Structure of the  $C_{11}H_{15}N_3O_3S$  molecule.

3-BENZYL-5-METHYLTHIO-3H,7H-1,2,3-TRIAZOLO[4,5-d][1,3]THIAZINE

$C_{12}H_{12}N_4S_2$

A. DUNAND, 1982. Acta Cryst., B38, 2299-2301.

Triclinic,  $P\bar{1}$ ,  $a = 8.742$ ,  $b = 9.717$ ,  $c = 9.823$  Å,  $\alpha = 110.09^\circ$ ,  $\beta = 113.70^\circ$ ,  $\gamma = 102.17^\circ$ ,  $D_m = 1.38$ ,  $Z = 2$ . Mo radiation,  $R = 0.051$  for 1469 reflexions.



In the molecule (Fig. 1) the 1,3-thiazine ring has a distorted boat-like conformation characterized by the torsion angles  $C(9)-N(4)-C(5)-S(6)$   $2.6(5)^\circ$  and  $S(6)-C(7)-C(8)-C(9)$   $-30.4(5)^\circ$ . The fused 1,2,3-triazole ring is planar.

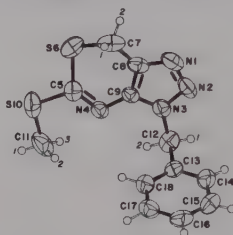


Fig. 1.  $C_{12}H_{12}N_4S_2$ : a perspective view of the molecule.

6-t-BUTYL-3-METHYL-5-THIOFORMYLPYRROLO[2,1-b]THIAZOLE  
 $C_{12}H_{15}NS_2$

A. SHARMA, J.L. LAWRENCE and R.C.G. KILLEAN, 1982. *Acta Cryst.*, **B38**, 962-963.

Monoclinic,  $P2_1/n$ ,  $a = 7.195$ ,  $b = 13.221$ ,  $c = 12.832$  Å,  $\beta = 94.65^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.031$  for 534 reflexions.

In the molecule (Fig. 1) the thioformyl group is in an anti position. The bond lengths of the thioformyl group and the pyrrole ring indicate that the molecule resonates between the covalent and the partly ionic forms.

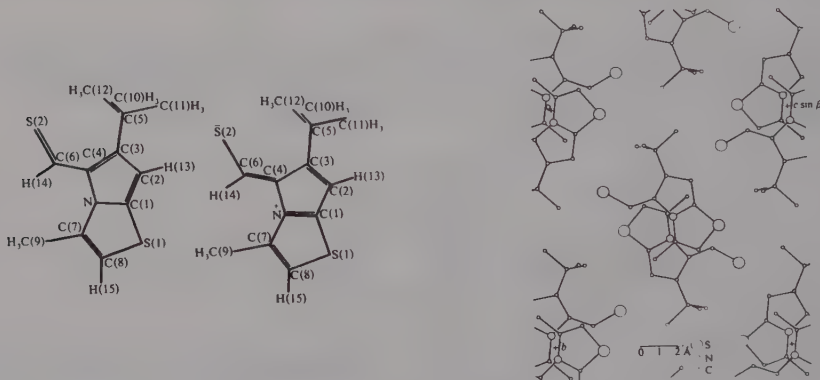


Fig. 1.  $C_{12}H_{15}NS_2$ : canonical forms of the molecule (left) and a view of the molecular packing down a (right).

3-PHENYLAZO-1H-4,1,2-BENZOTHIADIAZINE  
 $C_{13}H_{10}N_4S$

C. PAKAWATCHAI and A.H. WHITE, 1982. *Aust. J. Chem.*, **35**, 2157-2160.

Orthorhombic,  $P2_12_12_1$ ,  $a = 15.15$ ,  $b = 12.008$ ,  $c = 6.756$  Å,  $D_m = 1.36$ ,  $Z = 4$ . Mo radiation,  $R = 0.030$ .

The structure (Fig. 1) determined earlier (1) is confirmed. The non-aromatic hydrogen atom is bonded to N(22) ( $0.85(4)$  Å) and is also involved in an intermolecular hydrogen bond  $N(22)-H \cdots N(11')$   $2.27(4)$  Å,  $167(4)^\circ$ .

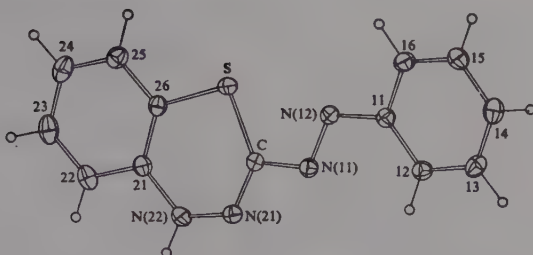


Fig. 1. A projection of the structure of 3-phenylazo-1H-4,1,2-benzothiadiazine.

1. W.S. McDONALD, H.M.N.H. IRVING, G. RAPER and D.C. RUPAINWAR, 1969. Chem. Commun., 392.

#### 1-METHYLPHENOTHIAZINE

$C_{13}H_{11}NS$  (I)

#### 1-ETHYLPHENOTHIAZINE

$C_{14}H_{13}NS$  (II)

S.S.C. CHU, V. NAPOLEONE, A.L. TERNAY Jr. and S. CHANG, 1982. Acta Cryst., B38, 2508-2511.

I. Monoclinic,  $P2_1/c$ ,  $a = 9.063$ ,  $b = 8.968$ ,  $c = 13.503$  Å,  $\beta = 102.29^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.053$  for 1658 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 9.509$ ,  $b = 8.962$ ,  $c = 14.036$  Å,  $\beta = 105.34^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.035$  for 1731 reflexions.

The crystal structures of I and II (Fig. 1) are nearly isomorphous. The molecules are folded with the central ring in a boat conformation; folding angles between the two benzo rings are  $154.8$  (I) and  $147.4^\circ$  (II). Bond lengths and angles are normal. There are no intermolecular contacts less than van der Waals distances.

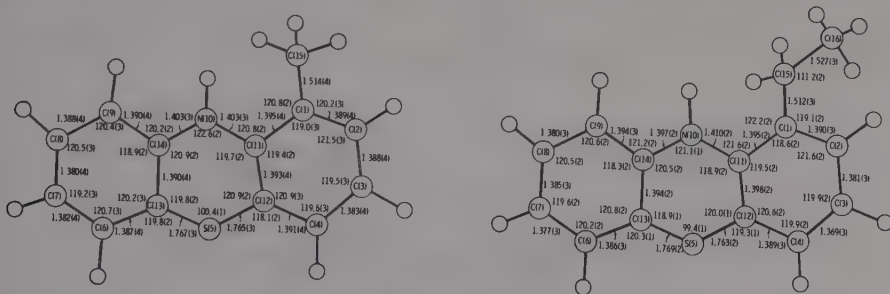


Fig. 1. Bond lengths and angles in  $C_{13}H_{11}NS$  (I) (left) and  $C_{14}H_{13}NS$  (II) (right).

#### 4-METHOXYCARBONYL-5-METHYL-2-PHENYL-6-THIA-2,3,8-TRIAZABICYCLO[3.2.1]OCT-3-ENE

$C_{13}H_{15}N_3O_2S$

J. GASTEIGER, U. STRAUZ and U. SCHUBERT, 1982. Chem. Ber., 115, 26-38.

Monoclinic,  $P2_1/c$ ,  $a = 9.725$ ,  $b = 16.34$ ,  $c = 8.334$  Å,  $\beta = 112.10^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.071$  for 1407 reflexions.

In the bicyclo moiety (Fig. 1) the five-membered ring adopts an envelope conformation with N(3) at the flap; the C(1),N(2),N(1),C(4),C(2),C(4) fragment is planar within experimental error. Principal bond lengths are: C(1)-N(2) 1.483(6), N(1)-N(2) 1.341(6), N(1)-C(4) 1.286(6), C(4)-C(5) 1.495(8), C(4)-C(2) 1.519(7), C(2)-N(3) 1.458(8), N(3)-C(1) 1.434(6) Å.

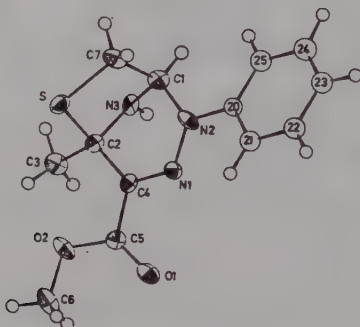


Fig. 1. Perspective view of  $C_{13}H_{15}N_3O_2S$ .

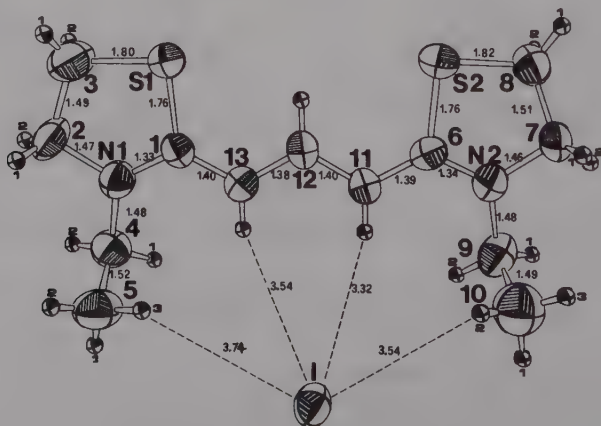
BIS(N-ETHYL-THIAZOLINE-2-YL)-TRIMETHINE-CYANINE IODIDE  
 $C_{13}H_{21}IN_2S_2$

$C_{13}H_{21}N_2S_2^+, I^-$

R. ALLMANN and S. OLEJNIK, 1982. Cryst. Struct. Comm., 11, 1175-1178.

Monoclinic,  $P2_1/n$ ,  $a = 12.896$ ,  $b = 16.615$ ,  $c = 7.974$  Å,  $\beta = 106.57^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.039$  for 2160 reflexions.

The cyanine cations (Fig. 1) are nearly parallel to the (001) plane. One thiazoline ring is in the half chair form (torsion angle N2-C7-C8-S2  $32.7^\circ$ ), whereas the other thiazoline ring is more planar (N1-C2-C3-S1  $-18.6^\circ$ ). But as indicated by the too short C2-C3 distance of 1.48 Å (uncorrected) and by the rather great  $B_{33}$  values (about perpendicular to the ring planes) of C2 and C3, this ring may oscillate between both possible half chair forms. Except for this small difference, the cyanine cation approximates a pseudo-mirror symmetry (through C12). The iodide anion is included in this pseudo-symmetry too as shown in Fig. 1 (H11...I 3.32, H13...I 3.54 Å). For previous work see 1.

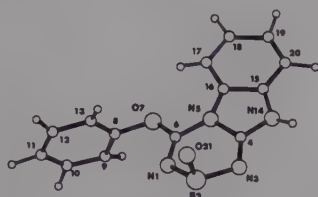


4-PHENOXY-10H-BENZIMIDAZO[2,1-c][1 $\lambda^4$ ,2,4,6]THIATRIAZINE 2-OXIDE  
 $C_{14}H_{10}N_4O_2S$

A. KÁLMÁN and G. ARGAY, 1982. *Acta Cryst.*, **B38**, 1370-1372.

Monoclinic,  $P2_1/n$ ,  $a = 9.329$ ,  $b = 4.603$ ,  $c = 30.124$  Å,  $\beta = 92.57^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.068$  for 1089 reflexions.

The molecule (Fig. 1) contains a rare trigonal-pyramidal  $N_2S=O$  moiety with an axially oriented O atom embedded in the non-planar thiatriazine ring. The  $S=O$  distance of 1.452 Å is considerably shorter than the 1.51 Å observed in various sulfoxides. The S-N distances are 1.705 and 1.669 Å. The molecules are linked in the crystal to form dimers by N-H...N hydrogen bonds with  $N...N$  2.864 Å.



N(1)-S(2)	1.705 (6)	C(8)-C(13)	1.334 (10)
N(1)-C(6)	1.247 (9)	C(9)-C(10)	1.407 (8)
S(2)-N(3)	1.669 (6)	C(10)-C(11)	1.360 (11)
S(2)-O(21)	1.452 (7)	C(11)-C(12)	1.374 (12)
N(3)-C(4)	1.286 (10)	C(12)-C(13)	1.408 (8)
C(4)-N(5)	1.415 (9)	N(14)-C(15)	1.378 (9)
C(4)-N(14)	1.359 (9)	C(15)-C(16)	1.413 (8)
N(5)-C(6)	1.385 (8)	C(15)-C(20)	1.352 (10)
N(5)-C(16)	1.404 (8)	C(16)-C(17)	1.372 (10)
C(6)-O(7)	1.350 (7)	C(17)-C(18)	1.374 (10)
O(7)-C(8)	1.434 (7)	C(18)-C(19)	1.393 (10)
C(8)-C(9)	1.400 (10)	C(19)-C(20)	1.368 (11)

Fig. 1.  $C_{14}H_{10}N_4O_2S$ : a perspective view of the molecule and bond lengths.

(3R,6R)-3-BENZYL-1,4-DIAZA-8-THIABICYCLO[4.4.0]DECANE-2,5-DIONE  
 $C_{14}H_{16}N_2O_2S$

M. VAN POUCKE and A.T.H. LENSTRA, 1982. *Cryst. Struct. Comm.*, **11**, 853-859.

Tetragonal,  $P4_12_12$ ,  $a = 10.019$ ,  $c = 27.156$  Å,  $Z = 8$ . Cu radiation,  $R = 0.049$  for 593 reflexions.

The analysis establishes the solid state conformation as shown in Fig. 1, with the six-membered sulphur-containing ring in chair form.

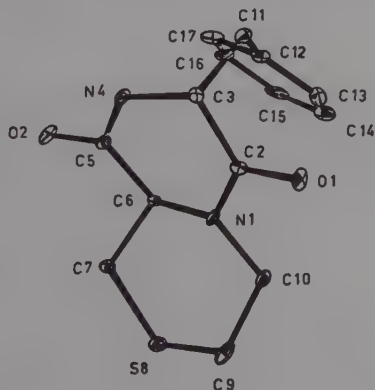


Fig. 1. The  $C_{14}H_{16}N_2O_2S$  conformation.

3-PHENYL-4-TOSYLIMINO-1,3-THIAZETIDINE-2-ONE  
 $C_{15}H_{12}N_2O_3S_2$

G.S.D. KING, L. VAN MEERVELT and G. L'ABBE, 1982. *Bull. Soc. Chim. Belg.*, **91**, 729-730.

Monoclinic,  $P2_1/n$ ,  $a = 29.091$ ,  $b = 6.171$ ,  $c = 8.327$  Å,  $\beta = 93.51^\circ$ ,  $D_m = 1.47$ ,  $Z = 4$ . Mo radiation,  $R = 0.031$  for 1225 reflexions.

Main bond lengths in the molecule (Fig. 1) are S1-C2 1.81, S1-C4 1.79, C2-N3 1.40, N3-C4 1.39, C2-O5 1.19, C4-N12 1.26, N12-S13 1.68. The internal ring angles are  $71.9$  (at S1),  $94.1$  (at C2),  $98.9$  (at N3) and  $95.0$  (C4). The S1...O15 distance is  $2.72$  Å, shorter than the sum of the van der Waals radii.

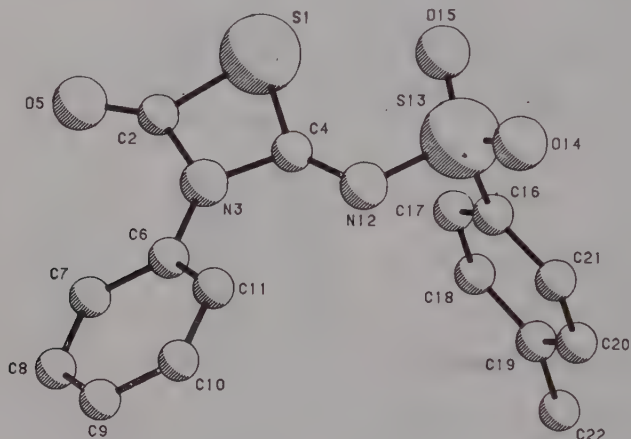


Fig. 1. The  $C_{15}H_{12}N_2O_3S_2$  structure.

4-HYDROXY-2-METHYL-N-2-PYRIDYL-2H-1 $\lambda$ <sup>6</sup>,2-BENZOTHAZINE-3-CARBOXAMIDE 1,1-DIOXIDE (PIROXICAM)

$C_{15}H_{13}N_3O_4S$

B. KOJIĆ-PRODIĆ and Ž. RUŽIĆ-TOROŠ, 1982. Acta Cryst., B38, 2948-2951.

Monoclinic,  $P2_1/c$ ,  $a = 7.127$ ,  $b = 15.136$ ,  $c = 13.949$  Å,  $\beta = 97.35^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.050$  for 2289 reflexions.

Bond lengths and angles in the molecule (Fig. 1) agree with expected values. The thiazine ring exhibits a half-chair conformation. An amide group is involved in an intramolecular hydrogen bond to the hydroxy group ( $O(17)-H(17)\dots O(15)$   $2.561(3)$  Å). It also forms an intermolecular hydrogen bond to an O atom bonded to the S atom ( $N(16)-H(16)\dots O(11)$   $3.053(3)$  Å), connecting piroxicam molecules in an infinite chain along  $b$ . The molecular packing is also influenced by van der Waals interactions.

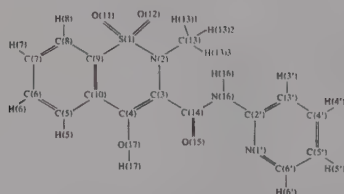
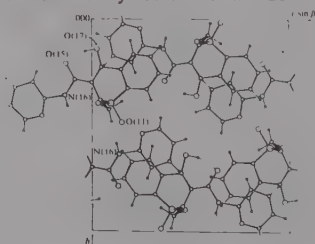


Fig. 1.  $C_{15}H_{13}N_3O_4S$ : structural formula of the molecule and a view of the molecular packing along  $a$ .



$$\text{C}_{15}\text{H}_{15}\text{NS}$$

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.716$ ,  $b = 8.366$ ,  $c = 19.963$  Å,  $Z = 4$ . Cu radiation,  $R = 0.059$  for 1139 reflections.

The molecule (Fig. 1) is folded with the central ring in a boat conformation; the folding angle between the two benzo rings is  $146.1^\circ$ . Bond lengths and angles are normal. There are no intermolecular contacts less than van der Waals distances.

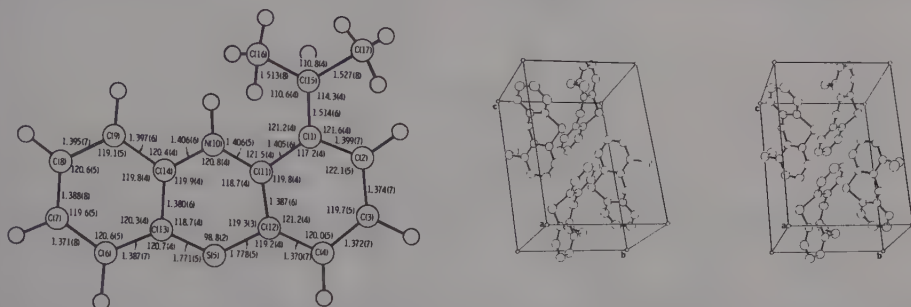


Fig. 1.  $C_{15}H_{15}NS$ : bond lengths and angles in the molecule and molecular packing in the crystal.

$$\text{C}_{16}\text{H}_{14}\text{N}_2\text{OS}$$

Monoclinic,  $P2_1/c$ ,  $a = 9.355$ ,  $b = 7.403$ ,  $c = 20.665$  Å,  $\beta = 87.14^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.043$  for 534 reflexions.

The molecule is a C-nitroso monomer (Fig. 1). There are no short, non-bonded contacts involving the nitroso group. The molecular dimensions are normal.

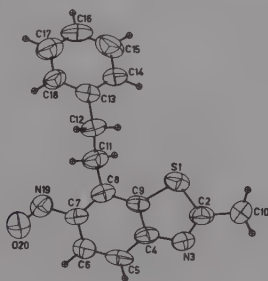


Fig. 1. The molecule of 2-methyl-6-nitroso-7-phenethyl-1,3-benzothiazole.

$$\text{C}_{17}\text{H}_{16}\text{N}_4\text{S}$$

B. BIRKNES and A. HORDVIK, 1982. Acta Chem. Scand., A36, 813-818.



Orthorhombic,  $Pbca$ ,  $a = 12.393$ ,  $b = 8.560$ ,  $c = 29.024$  Å,  $D_m = 1.34$ ,  $Z = 8$ . Cu radiation,  $R = 0.070$  for 2270 reflexions.

The central ring system is almost planar (Fig. 1) with bond distances  $N(1)-S(6a)$  1.895,  $N(6)-S(6a)$  1.873,  $N(1)-N(2)$  1.340,  $N(2)-C(3)$  1.330,  $C(3)-C(3a)$  1.398,  $C(3a)-S(6a)$  1.738,  $C(3a)-C(4)$  1.411,  $C(4)-N(5)$  1.351, and  $N(5)-N(6)$  1.329 Å. The  $N(1)-S(6a)-N(6)$  angle is  $167.4^\circ$ . The 1- and 6-phenyl groups are twisted 18 and  $15^\circ$  about the respective connecting bonds and the  $N(1)-C(15)-C(18)$  and  $N(6)-C(9)-C(12)$  vectors point to opposite sides of the central ring plane. The methyl C atoms  $C(7)$  and  $C(8)$  lie 0.09 and -0.13 Å from this plane.

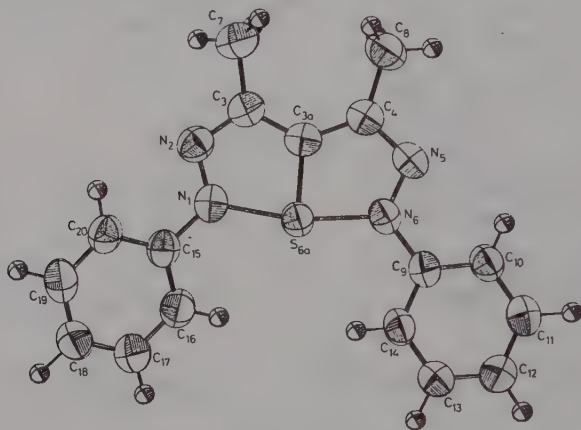


Fig. 1. Molecular structure of  $C_{17}H_{16}N_4S$ .

SODIUM 3-[(5-METHYL-1,3,4-THIADIAZOL-2-YL)THIOMETHYL]-8-OXO-7-[2-(2-AMINOTHIAZOL-4-YL)-(Z)-2-METHOXYIMINOACETAMIDOL-1-AZA-5-THIABICYCLO[4.2.0]OCT-2-EN-2-YL-CARBOXYLATE] MONOHYDRATE

$C_{17}H_{16}N_7NaO_5S_4 \cdot H_2O$

G. LAURENT, G. EVRARD and F. DURANT, 1982. *Eur. J. Med. Chem.-Chim. Ther.*, **17**, 281-284.

Monoclinic,  $P2_1$ ,  $a = 10.755$ ,  $b = 11.971$ ,  $c = 9.636$  Å,  $\beta = 95.14^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.123$  for 543 reflexions.

In spite of the low accuracy of the structure determination it is possible to see that the aminothiazole and methoxyimino groups are planar within experimental error (Fig. 1). Moreover, these two groups are quasi-coplanar (dihedral angle between the planes is  $2^\circ$ ), and nearly perpendicular to the exocyclic amide moiety. Estimated standard deviations on bond lengths and angles are respectively 0.1 Å and  $6^\circ$ , effectively preventing meaningful discussion of specific, as opposed to overall molecular geometry. There are intermolecular hydrogen bonds and bonding to the  $Na^+$  cation ( $Na(34) \cdots N(26)$  2.97,  $\cdots O(32')$  2.61 and  $\cdots O(35'')$  2.80 Å);  $N(30) \cdots O(20')$  2.87,  $N(30) \cdots O(9'')$  2.87,  $N(18) \cdots N(16''')$  2.97,  $N(15) \cdots O(35)$  3.00,  $O(33) \cdots O(35''')$  2.76 Å.

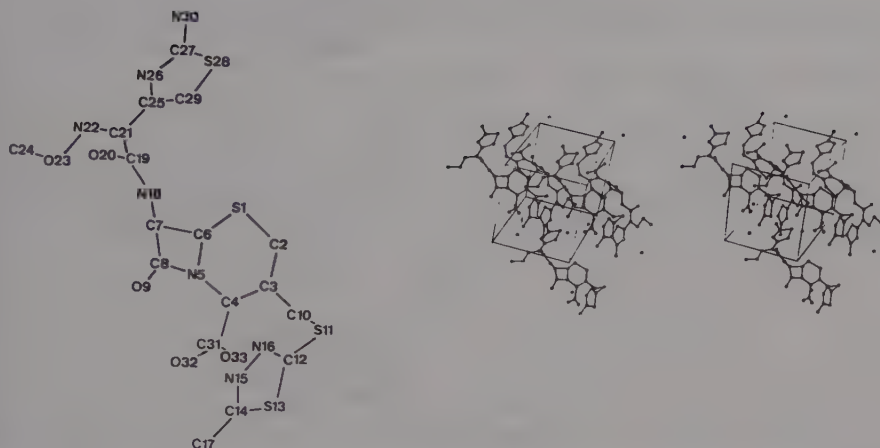


Fig. 1. Atomic labelling in  $C_{17}H_{16}N_7NaO_5S_4$  (left), and stereoview of molecular conformation and crystal packing (right).

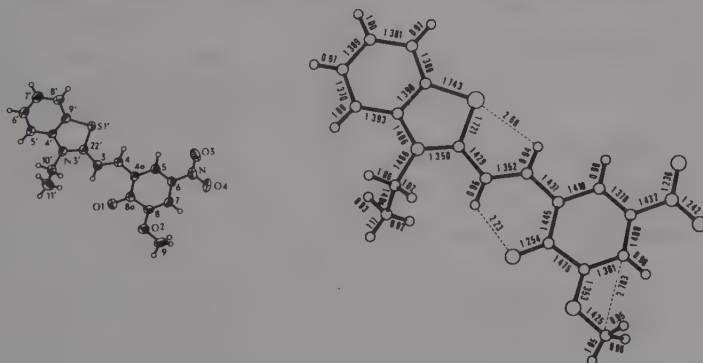
6-{2-(3-ETHYL-2,3-DIHYDRO-1,3-BENZOTHAZOL-2-YLIDENE)ETHYLIDENE}-2-METHOXY-4-NITRO-2,4-CYCLOHEXADIENEONE

$C_{18}H_{16}N_2O_4S$

E. MILER-SRENGER and R. GUGLIELMETTI, 1982. *Acta Cryst.*, B38, 1980-1984.

Monoclinic,  $P2_1/c$ ,  $a = 13.019$ ,  $b = 7.696$ ,  $c = 16.471$  Å,  $\beta = 91.95^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.062$  for 1701 reflexions.

In the solid state the polar, zwitterionic form is predominant - the geometry of the thiazole ring (Fig. 1) approaches that of a thiazolium cation and the phenolate ring is negatively charged. Except for C(5') and C(22') the atoms of the benzothiazole group are coplanar, the atoms C(22'), C(3), C(4) and C(4a) are coplanar while the phenolate ring is slightly non-planar (torsion angles about C(6)-C(7) and C(4a)-C(8a) are  $-3.4$  and  $-2.9^\circ$  respectively) with O(1) and NO<sub>2</sub> bent out on the same side of the plane. The dihedral angle between the benzothiazole and phenolate mean planes is  $5.2(5)^\circ$ . Molecules associate in pairs across a centre of symmetry such that alternating layers of phenolate and thiazole ring planes are stacked along  $b$ .



2,3,8,8a-TETRAHYDRO-5,8a-DIPHENYL-1H-IMIDAZO[2,1-c][1,4]THIAZINE  
 $C_{18}H_{18}N_2S$

S.S. SANDHU and M.S. HUNDAL, 1982. *Acta Cryst.*, B38, 842-844.

Monoclinic,  $P2_1/c$ ,  $a = 7.4729$ ,  $b = 23.5631$ ,  $c = 8.7006$  Å,  $\beta = 104.965^\circ$ ,  $D_m = 1.31$ ,  $Z = 4$ . Mo radiation,  $R = 0.0669$  for 1909 reflexions.

In the molecule (Fig. 1) S-C(sp<sup>2</sup>) and S-C(sp<sup>3</sup>) distances are 1.747 and 1.796 Å respectively. The C-S-C bond angle is  $97.8^\circ$  and the average C-C bond distance in the phenyl rings is 1.392 Å.

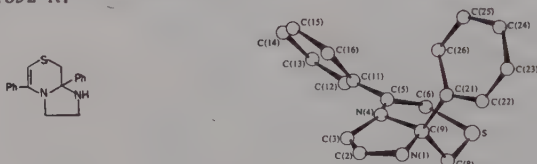


Fig. 1. The  $C_{18}H_{18}N_2S$  molecule viewed down the  $c$  axis.

9-(1-PIPERIDYL)THIOXANTHENE  
 $C_{18}H_{19}NS$

V. NAPOLEONE and S.S.C. CHU, 1982. *Cryst. Struct. Comm.*, 11, 621-626.

Orthorhombic,  $Pna2_1$ ,  $a = 17.655$ ,  $b = 5.483$ ,  $c = 15.548$  Å,  $Z = 4$ . Cu radiation,  $R = 0.044$  for 1257 reflexions.

The folding angle between the planes of the two benzene rings is  $135.6^\circ$  (Fig. 1); the central thioxanthene ring is in boat conformation and the piperidyl ring is in chair form. The torsion angle C(11)-C(9)-N(15)-C(20) is  $172.8^\circ$ .

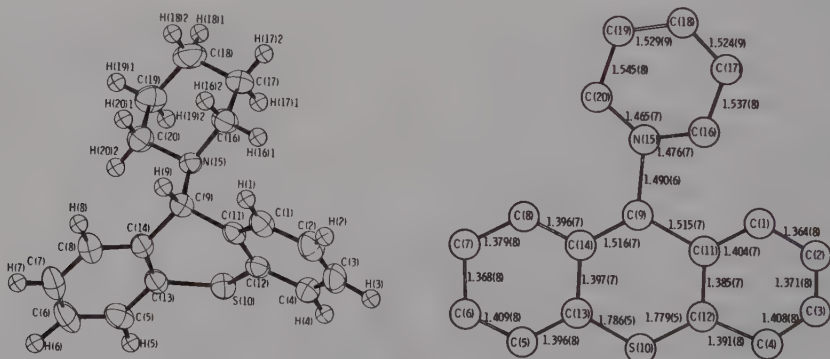


Fig. 1. The  $C_{18}H_{19}NS$  molecule and bond lengths.

AZOBENZENE N-(BENZOTHAZOL-2-YL)-IMIDE  
 $C_{19}H_{14}N_4S$

K.T. POTTS, A.J. ELLIOTT, G.R. TITUS, D. AL-HILAL, P.F. LINDLEY, G.V. BOYD and T. NORRIS, 1982. *Acta Cryst.*, B38, 682-685.

Triclinic,  $P\bar{1}$ ,  $a = 11.699$ ,  $b = 8.170$ ,  $c = 10.388$  Å,  $\alpha = 106.13$ ,  $\beta = 108.16$ ,  $\gamma = 106.76^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.044$  for 1817 reflexions.

The bond lengths (Fig. 1) in the molecule are consistent with a dipolar structure. In addition N(11) is only 0.043 Å from the N(10)N(12)C(111) plane. The S(1)...N(12) distance is 2.560(4) Å.

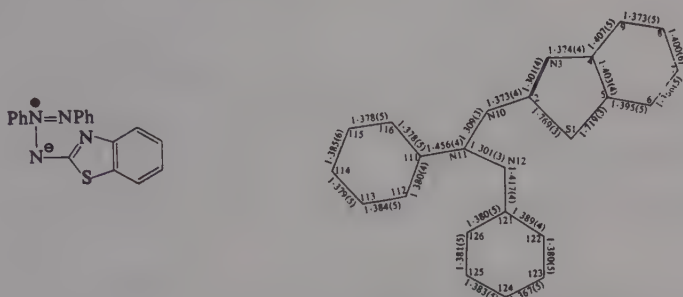


Fig. 1. Bond lengths in the  $C_{19}H_{14}N_4S$  molecule.

1,3-DIMETHYL-3',4'-DIPHENYLIMIDAZOLIDINE-2-SPIRO-2'(2'H)-THIETE-4,5-DIONE  
C<sub>19</sub>H<sub>16</sub>N<sub>2</sub>O<sub>2</sub>S (I)

1,3-DIMETHYL-4',5'-DIPHENYLIMIDAZOLIDINE-2-SPIRO-3'(3'H)-1',2'-DITHIOLE-4,5-DIONE  
C<sub>19</sub>H<sub>16</sub>N<sub>2</sub>O<sub>2</sub>S<sub>2</sub> (II)

1,3-DIMETHYL-2-(1,2-DIPHENYLETHYL) IMIDAZOLIDINE-4,5-DIONE  
C<sub>19</sub>H<sub>20</sub>N<sub>2</sub>O<sub>2</sub> (III)

H. GOTTHARDT and O.M. HUSS, 1982. Acta Cryst., B38, 875-880.

I. Orthorhombic,  $Pbca$ ,  $a = 14.164$ ,  $b = 12.766$ ,  $c = 18.736$  Å,  $D_m = 1.32$ ,  $Z = 8$ . Cu radiation,  $R = 0.047$  for 2131 reflexions.

II. Monoclinic,  $P2_1/n$ ,  $a = 8.352$ ,  $b = 9.267$ ,  $c = 23.285$  Å,  $\beta = 96.942^\circ$ ,  $D_m = 1.36$ ,  $Z = 4$ . Cu radiation,  $R = 0.038$  for 3041 reflexions.

III. Orthorhombic,  $Pbca$ ,  $a = 12.930$ ,  $b = 20.643$ ,  $c = 12.364$  Å,  $D_m = 1.25$ ,  $Z = 8$ . Cu radiation,  $R = 0.054$  for 1909 reflexions.

In all three molecules (Fig. 1) bond distances and angles are generally as expected, with the exception of a long S-C(sp<sup>iro</sup>) distance of 1.903 Å in I.

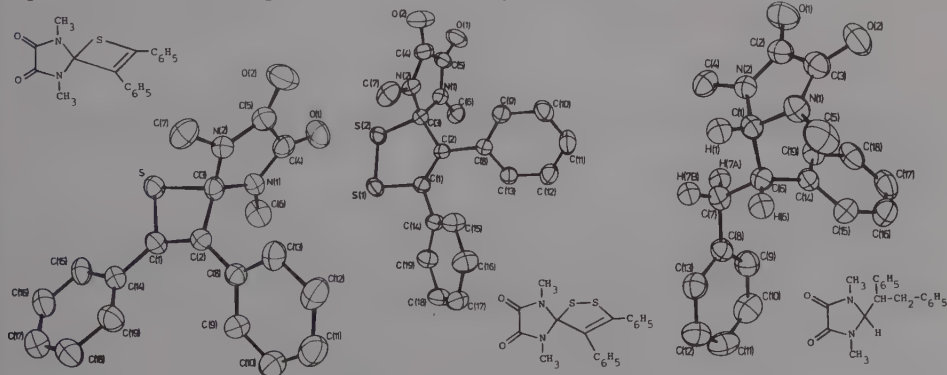
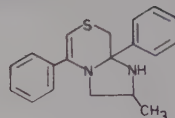


Fig. 1. Perspective views of the  $C_{19}H_{16}N_2O_2S$  (I) (left),  $C_{19}H_{16}N_2O_2S_2$  (II) (centre) and  $C_{19}H_{20}N_2O_2$  (III) molecules (right).

2,3,8,8a-TETRAHYDRO-2-METHYL-5,8a-DIPHENYL-1H-IMIDAZO[2,1-c][1,4]THIAZINE  
 $C_{19}H_{20}N_2S$



S.S. SANDHU and M.S. HUNDAL, 1982. *Acta Cryst.*, **B38**, 697-699.

Monoclinic,  $P2_1/n$ ,  $a = 16.980$ ,  $b = 12.430$ ,  $c = 7.800$  Å,  $\beta = 101.48^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.086$  for 1679 reflexions.

Some dimensions in the molecule are  $S-C(sp^2)$  1.740(9),  $S-C(sp^3)$  1.798(9) Å and  $C-S-C$  97.6(4)°.

3-THIA-7-AZA-6,8-DIPHENYLBICYCLO[3.3.1]NONAN-9-OL  
 $C_{19}H_{21}NOS$

E.L. ELIEL, M. MANOHARAN, D.J. HODGSON, D.S. EGGLESTON and R. JEYARAMAN, 1982. *J. Org. Chem.*, **47**, 4353-4356.

Monoclinic,  $C_m$ ,  $a = 7.222$ ,  $b = 20.544$ ,  $c = 5.829$  Å,  $\beta = 110.41^\circ$ ,  $D_m = 1.25$ ,  $Z = 2$ . Mo radiation,  $R = 0.059$  for 1120 reflexions.

The sulphur containing ring has a chair conformation and the nitrogen ring is in boat form (Fig. 1). The S atom sits 0.812 Å above, and C9 0.775(4) Å below, the C2,C4,C1,C5 plane while N and C9 are below the C8,C6,C1,C5 plane by 0.692 and 0.657(4) Å respectively. The dihedral angle between the plane defined by the phenyl ring and the C8-C6-C1-C15 plane is 105.3°.

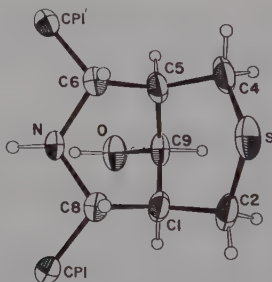


Fig. 1. A view of  $C_{19}H_{21}NOS$ . A crystallographic mirror plane passes through atoms N, O, C9 and S.

10-(3-DIMETHYLAMINO-2-METHYLPROPYL)-2-METHOXYPHENOTHIAZINE 5-OXIDE (LEVOMEPRAMAZINE SULPHOXIDE)

$C_{19}H_{24}N_2O_2S$

E. HOUGH, M. HJORTH and S.G. DAHL, 1982. *Acta Cryst.*, **B38**, 2424-2428.

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.636$ ,  $b = 12.783$ ,  $c = 18.317$  Å,  $Z = 4$ . Mo radiation,  $R = 0.037$  for 2529 reflexions (at 120 K). At 293 K  $a = 7.689$ ,  $b = 12.908$ ,  $c = 18.599$  Å,  $D_m = 1.223$ ,  $Z = 4$ . Mo radiation,  $R = 0.098$  for 933 reflexions.

The structure analysis was carried out initially at 293 K and then at 120 K. In the molecule the central thiazine ring is in the expected boat conformation and the sulphoxide oxygen atom O(15) is in an axial position with respect to this ring (Fig. 1). The N(10) side chain has the same conformation as that in chlorpromazine and several other psychoactive phenothiazine derivatives (1). Most of the bond lengths and angles are normal and intermolecular contacts correspond to van der

Waals distances.

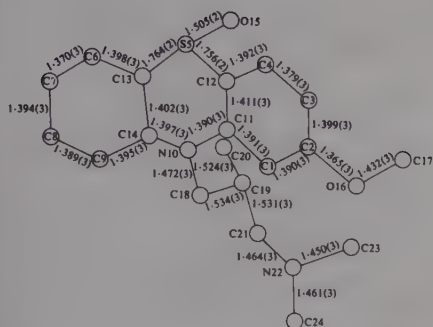


Fig. 1.  $C_{19}H_{24}N_2O_2S$ : bond lengths in the molecule and a stereoscopic view of the molecular packing, seen approximately down the  $b$  axis.

# 1. Structure Reports, 34B, 213.

2,2,7,7-TETRAMETHYL-4,9-DIPHENYL-1,6-DITHIA-3,8-DIAZASPIRO[4,4]NONA-3,8-DIENE  
 $C_{21}H_{22}N_2S_2$

D. OBRECHT, R. PREWO, J.H. BIERI and H. HEIMGARTNER, 1982. *Helv. Chim. Acta*, **65**, 1825-1836.

Triclinic,  $P\bar{1}$ ,  $a = 9.143$ ,  $b = 10.286$ ,  $c = 10.888$  Å,  $\alpha = 75.47^\circ$ ,  $\beta = 86.94^\circ$ ,  $\gamma = 69.74^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.035$  for 6716 reflexions (at  $-140^\circ C$ ).

The analysis established the structure as shown in Fig. 1.

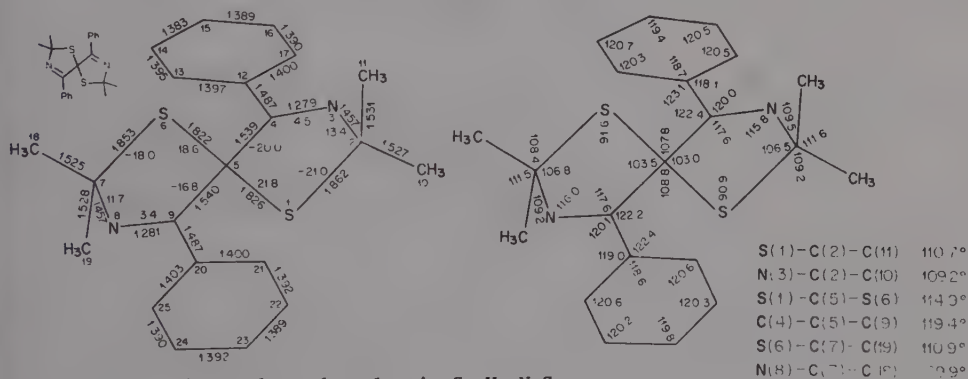


Fig. 1. Bond lengths and angles in  $C_{21}H_{22}N_2S_2$ .

METHYL 2-[(1R,3S,4R,5S,6S,8R)-3-BENZYL-4,5-BIS(METHOXYCARBONYL)-6-ETHOXYCARBONYL-9-  
 OXO-7,10-DIAZA-2-THIATRICYCLO[6.2.0.0<sup>3,7</sup>]DECAN-10-YL]-3-METHYLBUT-2-ENOATE  
 $C_{27}H_{32}N_2O_9S$

A.M. MARA, O. SINGH, E.J. THOMAS and D.J. WILLIAMS, 1982. *J. Chem. Soc. Perkin I*, 2169-2173.

Triclinic,  $P1$ ,  $a = 7.796$ ,  $b = 9.815$ ,  $c = 10.746$  Å,  $\alpha = 115.64^\circ$ ,  $\beta = 97.70^\circ$ ,  $\gamma = 101.37^\circ$ ,  $Z = 1$ . Cu radiation,  $R = 0.039$  for 2060 reflexions.



The bond lengths in the  $\beta$ -lactam ring (Fig. 1) are C(4)-C(5) 1.561, C(5)-N(6) 1.471, N(6)-C(7) 1.366 and C(4)-C(7) 1.536 Å, and the angles are C(4)-C(5)-N(6) 87.4, C(5)-N(6)-C(7) 95.1, N(6)-C(7)-C(4) 92.3 and C(7)-C(4)-C(5) 85.1°. The X-ray structure establishes the absolute configuration of the molecule.

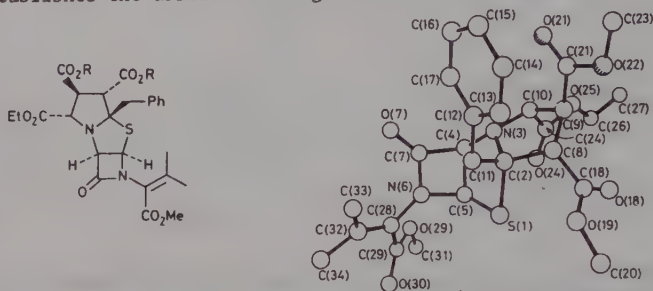


Fig. 1. Molecular skeleton (R = Me) and structure of  $C_{27}H_{32}N_2O_9S$ .

**trans-2,6-DIMETHYL-1,3-DIOXA-2,6-DIPHOSPHACYCLOOCTANE 2,6-DISULPHIDE**  
 $C_6H_{14}O_2P_2S_2$

C. PICCINNI-LEOPARDI, G. GERMAIN, J.P. DECLERCQ, M. VAN MEERSSCHE, J.B. ROBERT and K. JURKSCHAT, 1982. *Acta Cryst.*, B38, 2197-2199.

Monoclinic,  $P2_1/n$ ,  $a = 9.954$ ,  $b = 9.632$ ,  $c = 12.165$  Å,  $\beta = 102.31^\circ$ ,  $D_m = 1.40$ ,  $Z = 4$ . Mo radiation,  $R = 0.037$  for 1770 reflexions.

In the molecule (Fig. 1) the eight-membered ring adopts an asymmetric boat-chair conformation. Some bond lengths involving P and S are: P(2)-O(1) 1.594(2), P(2)-O(3) 1.589(2), P(2)-C(10) 1.793(3), P(2)-S(9) 1.918(1), P(6)-C(5) 1.821(2), P(6)-C(7) 1.828(2), P(6)-C(12) 1.800(3) and P(6)-S(11) 1.954(1) Å. Intermolecular contacts correspond to van der Waals interactions.

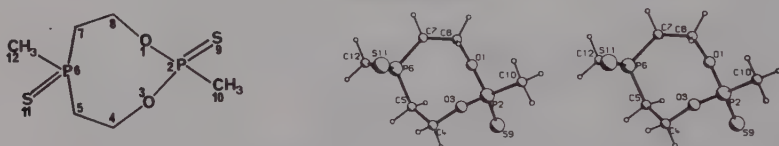


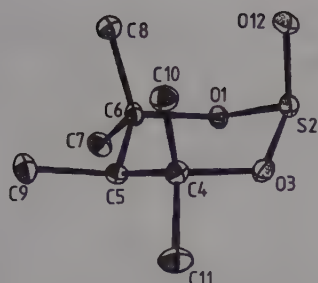
Fig. 1.  $C_6H_{14}O_2P_2S_2$ : structural formula and stereoscopic view of the molecule.

**cis-4,4,5,6,6-PENTAMETHYL-1,3,2-DIOXATHIAN-2-OXIDE**  
 $C_8H_{16}O_3S$

G.H. PETIT, A.T.H. LENSTRA and H.J. GEISE, 1982. *Cryst. Struct. Comm.*, 11, 867-872.

Monoclinic,  $P2_1/n$ ,  $a = 5.871$ ,  $b = 12.846$ ,  $c = 25.752$  Å,  $\beta = 95.35^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.047$  for 2600 reflexions.

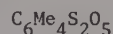
The two independent molecules have the same conformation (Fig. 1) with axial S=O.



	Mol A	Mol B		Mol A	Mol B
O1 - S2	1.599 (1)	1.603 (1)	O3 - S2	1.599 (1)	1.597 (1)
O1 - C6	1.487 (2)	1.483 (2)	O3 - C4	1.487 (2)	1.494 (2)
C4 - C5	1.550 (2)	1.541 (2)	C5 - C6	1.545 (2)	1.566 (2)
C4 - C10	1.525 (2)	1.514 (2)	C6 - C7	1.522 (2)	1.515 (2)
C4 - C11	1.508 (2)	1.513 (2)	C6 - C8	1.519 (2)	1.512 (2)
S2 - O12	1.452 (1)	1.447 (1)	C5 - C9	1.527 (2)	1.529 (2)

Fig. 1. The  $C_8H_{16}O_3S$  molecule and bond lengths.

3,4,5,6-TETRAMETHYL-1,2-BENZENEDISULPHONIC ANHYDRIDE  
 $C_{10}H_{12}S_2O_5$



J.D. SCHAGEN, P. SEIGNETTE and C.H. STAM, 1982. *Cryst. Struct. Comm.*, **11**, 1643-1645.

Monoclinic,  $P2_1/c$ ,  $a = 8.9528$ ,  $b = 13.575$ ,  $c = 10.7229$  Å,  $\beta = 113.94^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.039$  for 1635 reflexions.

The benzene ring (Fig. 1) is planar within 0.003 Å. The adjacent oxadithiole ring is an envelope with C(1), C(2), S(1) and S(2) coplanar within 0.007 Å and O(3) at a perpendicular distance of 0.294 Å, implying a folding angle of  $20.4^\circ$ . The C atoms of the methyl groups are only slightly out of the plane of the benzene ring (maximum distance 0.018 Å); S(1) and S(2), however, are at 0.121 and 0.086 Å respectively from this plane. As a result there is an angle of  $3.8^\circ$  between the benzene ring and the planar part of the oxadithiole ring in a direction opposite to the folding of the latter ring.

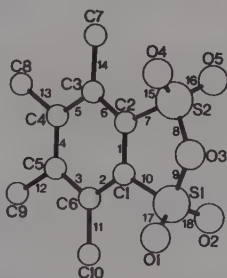


Fig. 1. A view of  $C_6Me_4S_2O_5$ .

1-NITROPHENOXATHIIN  
 $C_{12}H_7NO_3S$  (I)

9-NITRO-1-AZAPHENOXATHIIN  
 $C_{11}H_6N_2O_3S$  (II)

M.B. HOSSAIN, C.A. DWIGGINS, D. VAN DER HELM, P.K. SENGUPTA, J.C. TURLEY and G.E. MARTIN, 1982. *Acta Cryst.*, **B38**, 881-888.

I. Monoclinic,  $P2_1$ ,  $a = 13.864$ ,  $b = 7.160$ ,  $c = 10.950$  Å,  $\beta = 111.20^\circ$ ,  $D_m = 1.564$ ,  $Z = 4$ . Cu radiation,  $R = 0.033$  for 2250 reflexions (at 138 K).

II. Orthorhombic,  $Pbc2_1$ ,  $a = 3.799$ ,  $b = 19.940$ ,  $c = 25.874$  Å,  $D_m = 1.610$ ,  $Z = 8$ . Cu radiation,  $R = 0.082$  for 2057 reflexions (at 138 K).

The molecule (Fig. 1) is nearly planar; the dihedral angle between the two ring planes is  $3.3(3)^\circ$ . The S atom has the greatest displacement from the ring plane ( $0.11(2)$  Å).

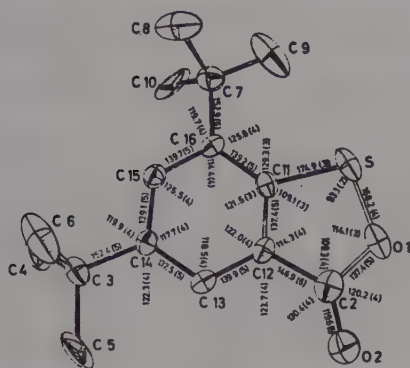


Fig. 1. The structure of  $C_{15}H_{20}O_2S$  with bond lengths (pm) and angles ( $^\circ$ ).

5,5'-BIS(DIMETHYLAMINO)-N,N,N',N'-TETRAMETHYL-(2,2'-BISELENAZOLE)-4,4'-DICARBOXAMIDE  
 $C_{16}H_{24}N_6O_2Se_2$

E. SCHAUAMANN, H. NIMMESGERN, G. ADIWIDJAJA and L. CARLSEN, 1982. Chem. Ber., 115, 2516-2525.

Monoclinic,  $P2_1/c$ ,  $a = 4.273$ ,  $b = 23.264$ ,  $c = 11.473$  Å,  $\beta = 120.41^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.100$  for 1528 reflexions.

Principal bond lengths in the centrosymmetric molecule (Fig. 1) are: C(2)-C(2') 1.44(2), Se-C(2,5) 1.87(1), C(2)-N(3) 1.28(1), N(3)-C(4) 1.39(1), C(4)-C(5) 1.37(1) Å. The selenazole ring is planar; the dihedral angles between this plane and those of the dimethylamino and dicarboxamide moieties are 15 and  $45^\circ$  respectively.

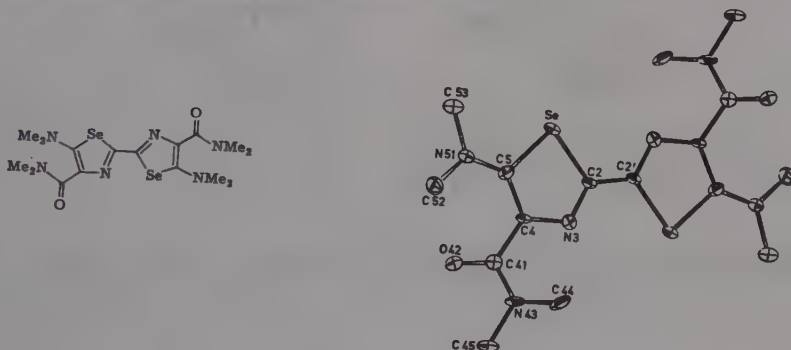


Fig. 1. The structure of  $C_{16}H_{24}N_6O_2Se_2$ .

(trans-F,trans-O,trans-C)-3,3,3',3'-TETRAKIS(TRIFLUOROMETHYL)-1,1'-SPIROBI[3H-2,1-BENZOXATHIOLE]-1,1-DIFLUORIDE  
 $C_{18}H_8F_{14}SO_2$  (I)

(cis-F,cis-O,trans-C)-3,3,3',3'-TETRAKIS(TRIFLUOROMETHYL)-1,1'-SPIROBI[3H-2,1-BENZOXATHIOLE]-1,1-DIFLUORIDE  
 $C_{18}H_8F_{14}SO_2$  (II)

R.S. MICHALAK and J.C. MARTIN, 1982. J. Am. Chem. Soc., 104, 1683-1692.

I. Triclinic,  $P\bar{1}$ ,  $a = 7.442$ ,  $b = 10.764$ ,  $c = 14.022$  Å,  $\alpha = 106.00^\circ$ ,  $\beta = 103.27^\circ$ ,  $\gamma = 105.00^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.047$  for 4041 reflexions.

II. Monoclinic,  $C2/c$ ,  $a = 18.800$ ,  $b = 7.641$ ,  $c = 16.599$  Å,  $\beta = 124.29^\circ$ ,  $D_m = 1.85$ ,  $Z = 4$ . Mo radiation,  $R = 0.037$  for 1265 reflexions.

Stereoviews of these geometrical isomers are shown in Fig. 1. The structure of the all trans isomer I consists of two essentially identical crystallographically independent molecules, each possessing exact  $C_i$  ( $\bar{1}$ ) symmetry. The coordination about S is octahedral in I, all angles being within  $1^\circ$  of the ideal values, and the five-membered  $C_3OS$  rings are planar to within  $0.007(3)$  Å. In II the coordination about S is also octahedral, the largest deviations from ideal geometry being in the O-S-O and F-S-F angles,  $93.91(9)$  and  $86.48(9)^\circ$ . Molecules of II possess exact  $C_2$  symmetry. The S-F, S-O, and S-C distances are:  $1.648(2)$ ,  $1.691(3)$ , and  $1.817(1)$  Å for I (mean values); and  $1.627(2)$ ,  $1.717(2)$ , and  $1.804(3)$  Å for II. The  $C_3OS$  rings in II are non-planar.

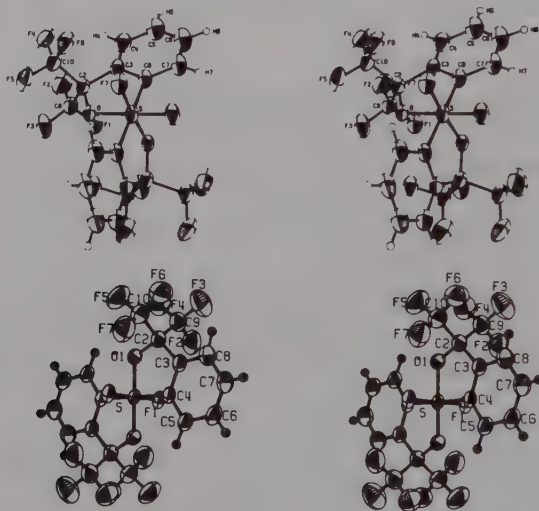
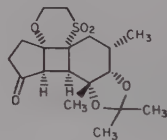


Fig. 1. Stereoviews of the all trans (top) and cis-F, cis-O, trans-C (bottom) isomers of  $C_{18}H_8F_{14}SO_2$ .

7,8-(ISOPROPYLIDENEDIOXY-0,0')-7,9-DIMETHYL-4,12,12-TRIOXO-5,12-OXATHIATRICYCLO-[9.4.0.0<sup>1,5</sup>.0<sup>6,11</sup>]PENTADECANE

$C_{18}H_{26}O_6S$



A.K. MUSSER and P.L. FUCHS, 1982. J. Org. Chem., 3121-3131.

Monoclinic,  $P2_1/n$ ,  $a = 12.958$ ,  $b = 10.064$ ,  $c = 15.388$  Å,  $\beta = 116.26^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.051$  for 2237 reflexions.

The analysis establishes the structure of this photoproduct as shown above.

3,6-BIS(DIMETHYLAMINO)-9,10-DIHYDRO-9-IODA-ANTHRACENE CHLORIDE  
 $C_{34}H_{40}ClI_2N_2$

HUANG ZHIYIN, CHEN YUANZHU and LU JIAXI, 1982. J. Struct. Chem., 1, No. 2, 77-82.

Monoclinic,  $P2_1/n$ ,  $a = 11.073$ ,  $b = 13.766$ ,  $c = 11.229$  Å,  $\beta = 99.36^\circ$ ,  $D_m = 1.60$ ,  $Z = 2$ . Mo radiation,  $R = 0.085$  for 2293 reflexions.

The centrosymmetric chloro-bridged dimer has the structure shown in Fig. 1. Some dimensions are mean C-I 2.127 Å, dihedral angle between pairs of adjacent phenyl rings is  $133^\circ$ .

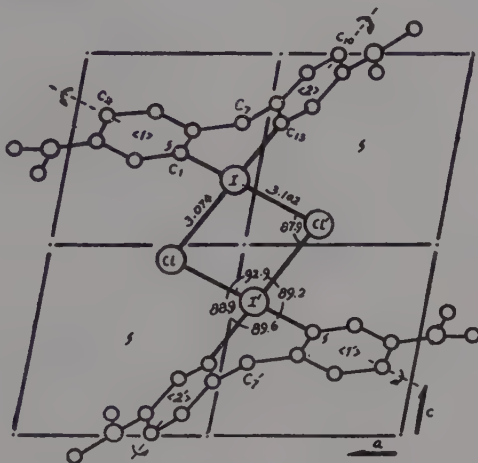
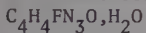


Fig. 1. A view of the  $C_{34}H_{40}ClI_2N_2$  molecule.

5-FLUOROCYTOSINE HYDRATE



T. LOUIS, J.N. LOW and P. TOLLIN, 1982. Cryst. Struct. Comm., 11, 1059-1064.

Monoclinic,  $P2_1/c$ ,  $a = 7.562$ ,  $b = 9.390$ ,  $c = 21.361$  Å,  $\beta = 125.13^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.076$  for 1060 reflexions.

There are two independent molecules with similar dimensions and they are almost coplanar (Fig. 1). Molecules are linked by hydrogen bonds and form continuous ribbons parallel to  $b$ ; the water molecules link the ribbons.



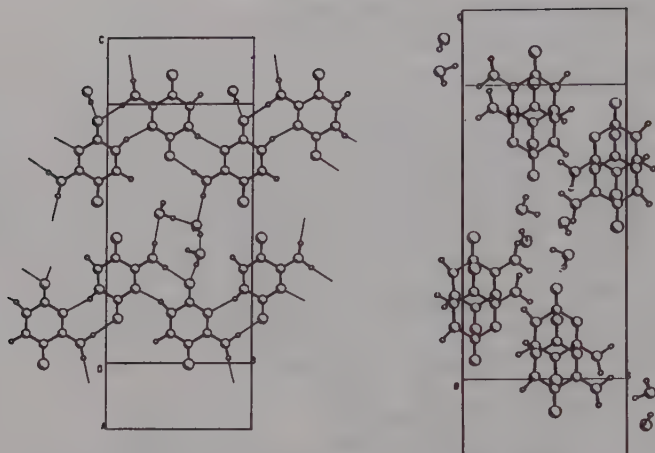


Fig. 1. Molecular packing of  $C_4H_4FN_3O, H_2O$

SODIUM  $N^4$ -HYDROXY-5,6-DIHYDROCYTOSINE-6-SULPHONATE MONOHYDRATE

$C_4H_6N_3NaO_5S, H_2O$  (I)

$C_4H_6N_3O_5S^-.Na^+, H_2O$

SODIUM  $N^4$ -HYDROXY-1-METHYL-5,6-DIHYDROCYTOSINE-6-SULPHONATE TETRAHYDRATE

$C_5H_8N_3NaO_5S, 4H_2O$  (II)

$C_5H_8N_3O_5S^-.Na^+, 4H_2O$

C.L. BARNES and S.W. HAWKINSON, 1982. *Acta Cryst.*, B38, 1907-1911.

I. Monoclinic,  $P2_1/c$ ,  $a = 12.977$ ,  $b = 8.384$ ,  $c = 8.552$  Å,  $\beta = 106.89^\circ$ ,  $D_m = 1.87$ ,  $Z = 4$ . Mo radiation,  $R = 0.036$  for 2325 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 6.491$ ,  $b = 11.129$ ,  $c = 17.883$  Å,  $\beta = 93.50^\circ$ ,  $D_m = 1.65$ ,  $Z = 4$ . Mo radiation,  $R = 0.037$  for 2624 reflexions.

Both compounds are in the imino form with the  $N^4$ -OH syn to  $N(3)$  (Fig. 1). The sulphonate groups are axial to the puckered rings with O atoms staggered with respect to the substituents on C(6). C(5) and C(6) are displaced 0.193 and 0.338 Å (in I) and 0.459 and 0.164 Å (in II) on opposite sides of the  $N(1), C(2), N(3), C(4)$  mean plane. In I layers of anions alternate with channels of solvent molecules and  $Na^+$  ions along a linked by hydrogen bonds. In II anions form discrete, centrosymmetric, hydrogen-bonded pairs. Adjacent pairs are bridged by hydrogen bonds to water molecules and by coordination to  $Na^+$ .

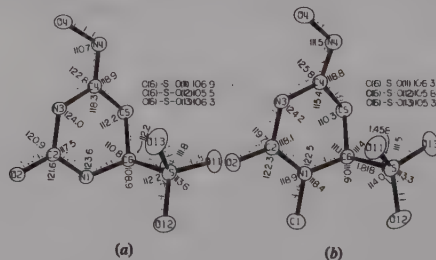


Fig. 1. Bond distances and angles in (a)  $C_4H_6N_3O_5S^-$  and (b)  $C_5H_8N_3O_5S^-$ .

## 2,4,6-TRIAMINOPYRIMIDINE



C.H. SCHWALBE and G.J.B. WILLIAMS, 1982. *Acta Cryst.*, **B38**, 1840-1843.

Monoclinic,  $P2_1/n$ ,  $a = 10.348$ ,  $b = 9.551$ ,  $c = 12.464 \text{ \AA}$ ,  $\beta = 112.27^\circ$ ,  $D_m = 1.45$ ,  $Z = 8$ . Cu radiation,  $R = 0.107$  for 995 reflexions.

The two independent molecules are very nearly planar (Fig. 1) but deviate by up to  $0.03(1) \text{ \AA}$  from the expected local  $C_{2v}$  symmetry. Molecules are linked into ribbons in the  $c$ -direction by N-H...N bonds joining the 4- and 6-amino groups to the ring N atoms. Two ribbons associate by  $\pi$ - $\pi$  interactions between equivalent molecules to form an infinite, antiparallel, double ribbon.

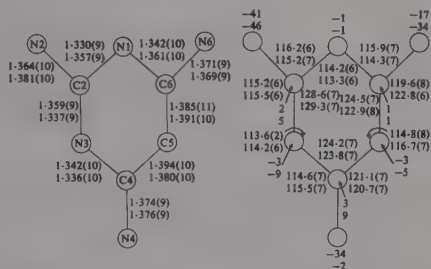
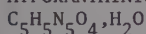


Fig. 1. Molecular geometry and displacements ( $\text{\AA} \times 10^3$ ) from the mean ring plane for both independent molecules in 2,4,6-triaminopyrimidine.

## HYPOXANTHINUM NITRATE MONOHYDRATE



R.D. ROSENSTEIN, M. OBERDING, J.R. HYDE, J. ZUBEITA, K.D. KARLIN and N.C. SEEMAN, 1982. *Cryst. Struct. Comm.*, **11**, 1507-1513.

Orthorhombic,  $Pnma$ ,  $a = 13.701$ ,  $b = 6.236$ ,  $c = 10.078 \text{ \AA}$ ,  $Z = 4$ .  $R = 0.050$  for 253 reflexions.

The cation, anion and water molecule lie on a crystallographic mirror plane (Fig. 1), and are interlinked by N-H...O and O-H...N hydrogen bonds. Nitrate ions stack one above the other, but hypoxanthinium cations are separated by water molecules which sandwich between them.

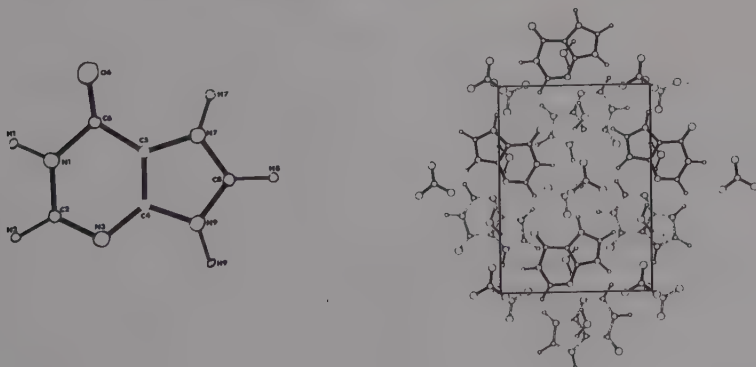


Fig. 1. The hypoxanthinium cation and a  $b$ -axis projection of the crystal structure.



## 3-(2,4-DIOXO-1-PYRIMIDINYL)PROPIONIC ACID (1-(2-CARBOXYETHYL)URACIL)



S. FUJITA, A. TAKENAKA and Y. SASADA, 1982. *Acta Cryst.*, B38, 2936-2938.

Monoclinic,  $P2_1/c$ ,  $a = 8.455$ ,  $b = 14.235$ ,  $c = 6.825$  Å,  $\beta = 110.12^\circ$ ,  $D_m = 1.58$ ,  $Z = 4$ . Cu radiation,  $R = 0.044$  for 1212 reflexions.

In the molecule (Fig. 1) the pyrimidine ring is planar and bond lengths and angles are generally as expected. The uracil moieties are paired around an inversion center through  $N(3)-N\cdots O(4)$  hydrogen bonds ( $N\cdots O$  2.825(2) Å) and the carboxyl group is hydrogen-bonded to  $O(2)$  of the uracil moiety in the neighbouring molecule ( $O\cdots O$  2.710(2) Å). This hydrogen bond may be one of the elementary interaction patterns between the carboxyl group of an acidic amino acid residue and the base-paired uracil.

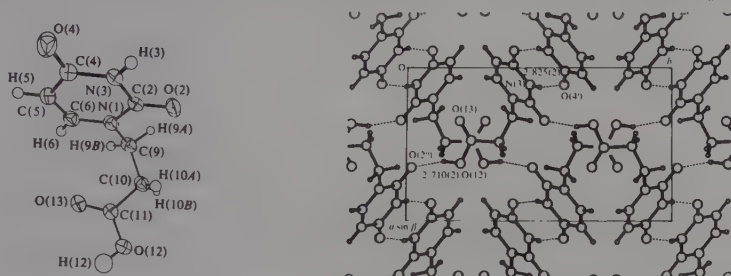
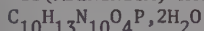


Fig. 1.  $\text{C}_7\text{H}_8\text{N}_2\text{O}_4$ : molecular structure and c-axial projection of the crystal structure.

## BIS(ADENINIUM) HYDROGEN PHOSPHATE DIHYDRATE



R.J. WALKER, P. TOLLIN and J.N. LOW, 1982. *Cryst. Struct. Comm.*, 11, 579-583.

Triclinic,  $P1$ ,  $a = 11.42$ ,  $b = 11.67$ ,  $c = 7.90$  Å,  $\alpha = 75.9^\circ$ ,  $\beta = 55.6^\circ$ ,  $\gamma = 85.9^\circ$ ,  $Z = 1$ . Mo radiation,  $R = 0.115$  for 328 reflexions.

The intramolecular short contacts (Fig. 1) suggest that the structure is held together by a network of hydrogen bonds. The analysis is of low accuracy because of the paucity of data.

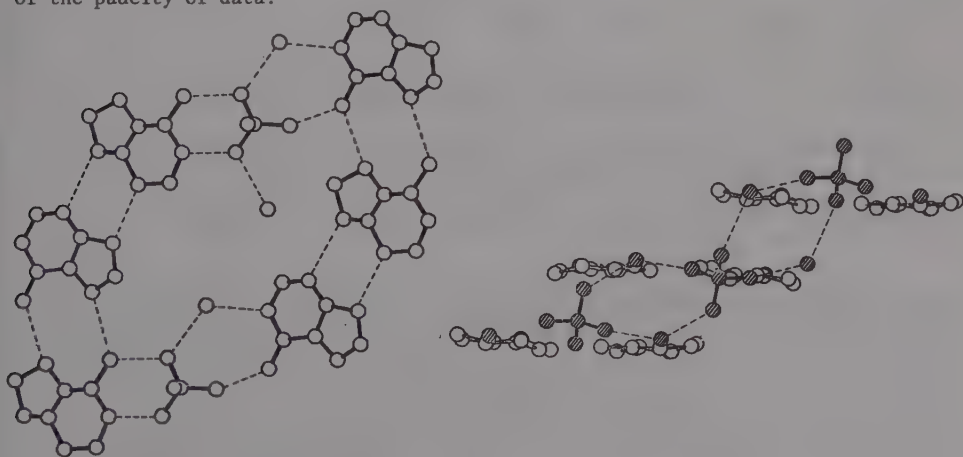


Fig. 1. Two views of  $\text{C}_{10}\text{H}_{13}\text{N}_{10}\text{O}_4\text{P}\cdot 2\text{H}_2\text{O}$ .

2,2,8,9-TETRAMETHYL-1,2-DIHYDROPURINE-6-CARBOXAMIDE  
 $C_{10}H_{15}N_5O$

B. BEAGLEY, B.L. BOOTH, R.G. PRITCHARD and M.F. PROENCA, 1982. *Acta Cryst.*, **B38**, 2921-2924.

Monoclinic,  $C2/c$ ,  $a = 13.900$ ,  $b = 8.992$ ,  $c = 18.091$  Å,  $\beta = 98.98^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.037$  for 1243 reflexions.

In the molecule (Fig. 1) the 1,2-dihydropurine ring system approaches planarity except for C(2) where the full ring conjugation found in purines is broken. The carbamoyl group is nearly coplanar with the conjugated ring atoms and forms an internal hydrogen bond to N(7). An interpenetrating network of hydrogen bonds links dimers  $(N(1)...O(12'))$   $3.106(3)$  Å through N(3) and N(11).

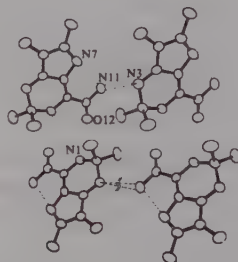
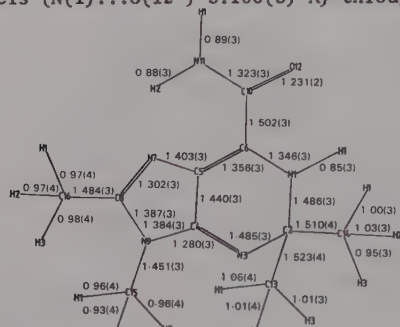


Fig. 1.  $C_{10}H_{15}N_5O$ : bond lengths in the molecule and a view indicating hydrogen bonding.

2-(METHYLAMINO)-6-CHLORO-4-(N-METHYLPIPERAZINO)-5-(METHYLTHIO)PYRIMIDINE  
 $C_{11}H_{18}ClN_5S$  (I)

2-(ISOPROPYLAMINO)-6-CHLORO-4-(N-METHYLPIPERAZINO)-5-(METHYLTHIO)PYRIMIDINE  
 $C_{13}H_{22}ClN_5S$  (II)

2-(BENZYLAMINO)-6-CHLORO-4-(N-METHYLPIPERAZINO)-5-(METHYLTHIO)PYRIMIDINE  
 $C_{17}H_{22}ClN_5S$  (III)

C. GUÉRÉMY, F. AUDIAU, A. UZAN, G. LE FUR, J.-M. LÉGER and A. CARPY, 1982. *J. Med. Chem.*, **25**, 1459-1465.

I. Triclinic,  $P\bar{1}$ ,  $a = 7.256$ ,  $b = 8.809$ ,  $c = 11.900$  Å,  $\alpha = 103.40^\circ$ ,  $\beta = 106.16^\circ$ ,  $\gamma = 86.00^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.041$  for 2559 reflexions.

II. Monoclinic,  $P2_1/n$ ,  $a = 13.987$ ,  $b = 10.810$ ,  $c = 10.563$  Å,  $\beta = 90.23^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.051$  for 1710 reflexions.

III. Monoclinic,  $P2_1/c$ ,  $a = 11.137$ ,  $b = 5.496$ ,  $c = 32.225$  Å,  $\beta = 65.85^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.036$  for 2649 reflexions.

In all three molecules the piperazine ring is in a chair conformation with an almost exact mirror plane through N(7) and N(10) (Fig. 1). The pyrimidine ring is equatorially oriented relative to the piperazine ring with the lone pairs on N(7) and N(10) orthogonal to one another in I. In II and III it is in an axial orientation and the lone pairs are cis to one another. The dihedral angle between the plane of the pyrimidine and the basic plane of the piperazine rings is  $149^\circ$  in I,  $100^\circ$  in II, and  $97^\circ$  in III. The position of the pyrimidine ring with respect to that of the piperazine ring is defined by a torsion angle  $\phi = C(6)-C(1)-N(7)-C(8)$  which is  $-171^\circ$  in I,  $-173^\circ$  in III and  $-166^\circ$  in II. The position of the  $S-CH_3$  group is characterized by the two torsion angles  $N(4)-C(5)-C(6)-S(13)$  and  $C(5)-C(6)-S(13)-C(14)$ .

which are 170 and  $-120^\circ$  in I, 173 and  $-67^\circ$  in II, and 170 and  $80^\circ$  in III. Finally, the position of the  $R_1$  group is defined by a torsion angle  $\psi = N(4)-C(3)-N(16)-C(17)$  which is  $178^\circ$  in I,  $179^\circ$  in II, and  $172^\circ$  in III.

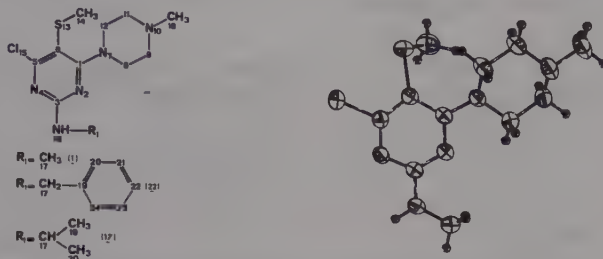


Fig. 1. Numbering scheme for molecules and the molecular structure of  $C_{11}H_{18}ClN_5S$ .

#### THIAMINE DICHLORIDE MONOHYDRATE



I.H. SUH, Y.-J. KIM, M.-J. KIM, Y.K. YOON and S.T. AHN, 1982. J. Korean Phys. Soc., 15, 114-121.

Monoclinic,  $P2_1/n$ ,  $a = 6.975$ ,  $b = 20.555$ ,  $c = 11.727$  Å,  $\beta = 98.78^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.097$  for 1602 reflexions.

Both the pyrimidine and thiazolium rings are planar within experimental error (Fig. 1) and make a dihedral angle with each other of  $76.3^\circ$ . They are turned so as to bring the amino group (N(18)) closer to the hydrogen-bearing carbom atom (C(2)) of the thiazolium ring. There are four intramolecular hydrogen bonds (O(9)-H(40)...O(19) 2.936 Å,  $136.8^\circ$ ; O(9)-H(22)...Cl(20) 3.201 Å,  $140.7^\circ$ ; N(18)-H(39)...Cl(20) 3.232 Å,  $159.2^\circ$ ; O(19)-H(41)...Cl(21) 3.060 Å,  $144.3^\circ$ ), and four intermolecular hydrogen bonds (N(18)-H(38)...Cl(20') 3.245 Å,  $175.8^\circ$ ; C(2)-H(30)...Cl(20'') 3.464 Å,  $158.6^\circ$ ; N(11)-H(34)...Cl(21') 3.034 Å,  $168.8^\circ$ ; C(16)-H(33)...Cl(21'') 3.511 Å,  $151.1^\circ$ ). The symmetry codes for the acceptor atoms in the intermolecular hydrogen bonds are respectively 1-x, 1-y, 1-z; 1+x, 0.5+y, 1.5-z; 2.5-x, 0.5+y, 1.5-z; 0.5+x, 0.5-y, 0.5+z.

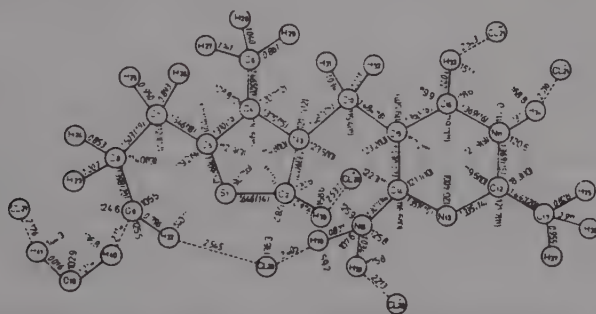


Fig. 1. Schematic representation of  $C_{12}H_{18}Cl_2N_4OS \cdot H_2O$  showing bond distances and angles (Å and degrees, respectively).



2-DIMETHYLAMINO-6-DIETHYLENEIMINOPHOSPHAMIDO-7-METHYLPURINE [FOPURINE]  
 $C_{12}H_{19}N_8OP$  (I)

2-DIMETHYLAMINO-6-AMINO-7-METHYLPURINE  
 $C_8H_{12}N_6$  (II)

V.N. VASIL'CHENKO, V.V. MITKEVICH, A.A. MOISEENKO, V.G. KHOMENKO and V.A. CHERNOV,  
 1982. Zh. Strukt. Khim., 23-2, 107-113 [J. Struct. Chem., 23, 257-263].

I. Triclinic,  $P\bar{1}$ ,  $a = 8.793$ ,  $b = 8.159$ ,  $c = 11.002$  Å,  $\alpha = 96.52^\circ$ ,  $\beta = 91.64^\circ$ ,  $\gamma = 101.38^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.088$  for 1035 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 6.871$ ,  $b = 19.678$ ,  $c = 8.362$  Å,  $\beta = 123.85^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.051$  for 790 reflexions.

Molecules of I and II are shown in Fig. 1. The geometry of the purine moieties in the two molecules is similar. Bond lengths ( $\sigma = 0.007$ - $0.02$  Å in I,  $0.006$ - $0.010$  Å in II) are as expected.

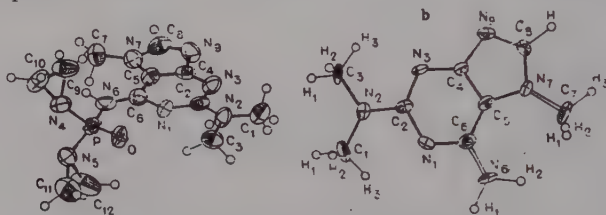


Fig. 1. The  $C_{12}H_{19}N_8OP$  (a) and  $C_8H_{12}N_6$  (b) molecules.

2-(2-PROPOXYPHENYL)-8-AZAHYPOXANTHINE  
 $C_{13}H_{13}N_5O_2$  (I)

2-(2-PROPOXY-5-(PROPYLSULFONYL)PHENYL)-8-AZAHYPOXANTHINE METHANOL  
 $C_{16}H_{19}N_5O_4S, CH_4O$

2-(2-PROPOXY-5-(N-METHYL-N-ISOPROPYLSULPHAMOYL)PHENYL)-8-AZAHYPOXANTHINE  
 $C_{17}H_{22}N_6O_4S$  (III)

S.R. WILSON, R.B. WILSON, A.L. SHOEMAKER, K.R.H. WOOLDRIDGE and D.J. HODGSON, 1982.  
 J. Am. Chem. Soc., 104, 259-264.

I. Orthorhombic,  $Pnma$ ,  $a = 9.293$ ,  $b = 6.671$ ,  $c = 21.020$  Å,  $D_m = 1.37$ ,  $Z = 4$ . Mo radiation,  $R = 0.043$  for 1029 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 12.521$ ,  $b = 5.816$ ,  $c = 28.388$  Å,  $\beta = 108.21^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.105$  for 918 reflexions.

III. Monoclinic,  $P2_1/c$ ,  $a = 12.129$ ,  $b = 10.680$ ,  $c = 16.958$  Å,  $\beta = 116.74^\circ$ ,  $D_m = 1.38$ ,  $Z = 4$ . Mo radiation,  $R = 0.050$  for 1771 reflexions.

The three molecules are shown in Fig. 1. In I and II, the entire ring systems are held approximately planar (exactly planar in the former case) by a

The three molecules are shown in Fig. 1. In I and II, the entire ring systems are held approximately planar (exactly planar in the former case) by a strong N(1)-H...O(2) intramolecular hydrogen bond, where O(2) is the oxygen atom of the propoxy group. In III, the purine and the phenyl ring are inclined at an angle of  $52.6^\circ$ . I and III are present as the N(9)-H tautomers, but II exhibits the N(8)-H tautomer. Bond lengths ( $\sigma = 0.003$ - $0.004$ ,  $0.01$ - $0.04$ , and  $0.003$ - $0.009$  Å for I, II and III respectively) and bond angles assume normal values.

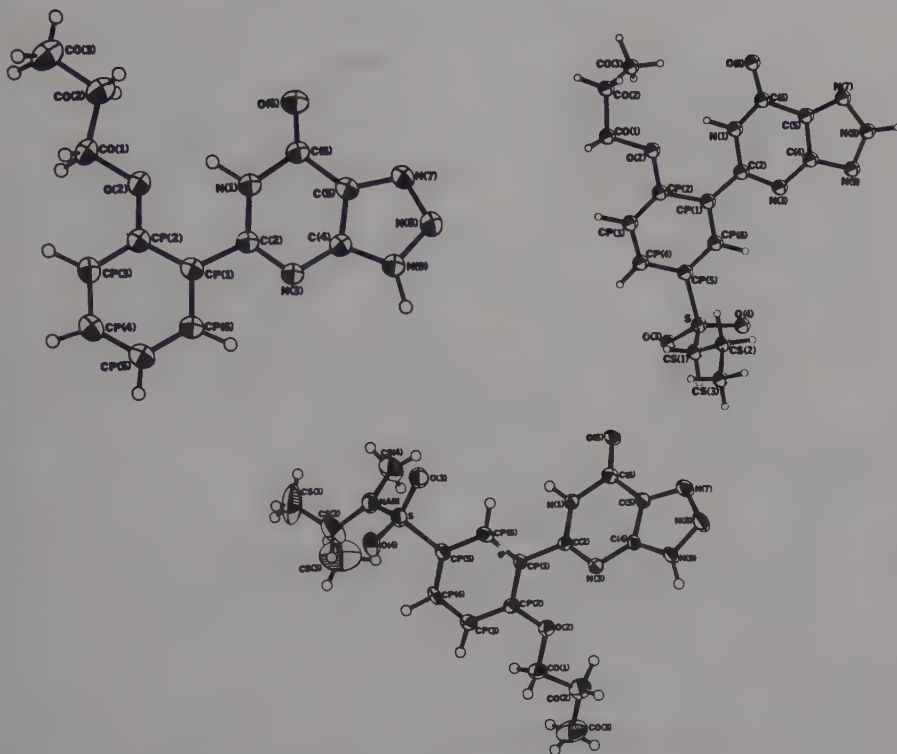


Fig. 1. The molecular structures of  $C_{13}H_{13}N_5O_2$  (I, top left),  $C_{16}H_{19}N_5O_4S$  (II, top right), and  $C_{17}H_{22}N_6O_4S$  (bottom).

1-[2-(ADENIN-9-YL)ETHYL]-3-CARBAMOYLPYRIDINIUM CHLORIDE MONOHYDRATE

$C_{13}H_{14}ClN_7O_2 \cdot H_2O$

$C_{13}H_{14}N_7O^+, Cl^-, H_2O$

T. ISHIDA, Y. MIYAMOTO, A. NAKAMURA and M. INOUE, 1982. *Acta Cryst.*, B38, 192-195.

Monoclinic,  $P2_1/a$ ,  $a = 12.073$ ,  $b = 8.280$ ,  $c = 15.086$  Å,  $\beta = 94.16^\circ$ ,  $D_m = 1.488$ ,  $Z = 4$ .  
Cu radiation,  $R = 0.078$  for 2622 reflexions.

The molecule (Fig. 1) has a folded conformation although there is no prominent overlap between the adenine and pyridinium rings. In the crystal (Fig. 1), the molecules are held together by a network of O-H...N, N-H...O, N-H...N and N-H...Cl hydrogen bonds.

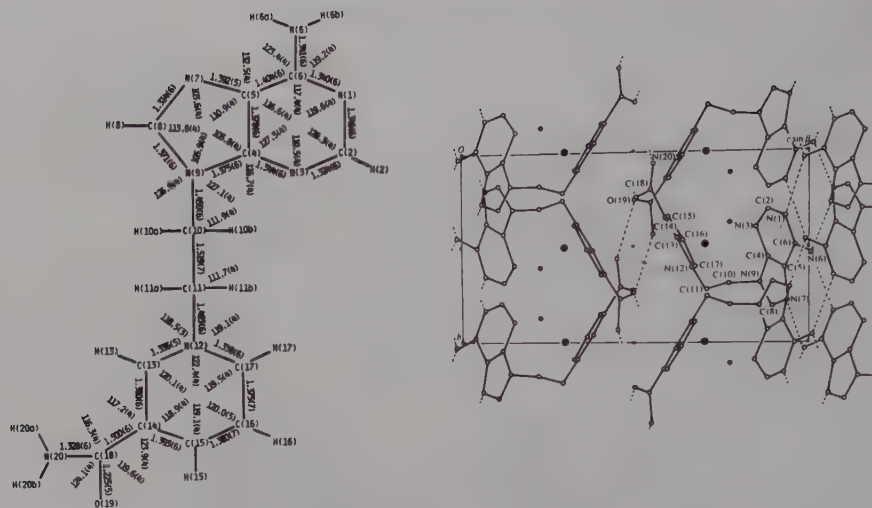


Fig. 1. Bond lengths and angles in the  $C_{13}H_{14}N_7O^+$  cation and a view of the crystal structure down the  $a$  axis.

cis-5-HYDROXY-1,3-DIMETHYL-6-PHENYLTHIO-5,6-DIHYDROTHYMINE  
 $C_{13}H_{16}N_2O_3S$  (I)

cis-5-HYDROXY-6-(p-HYDROXYPHENYL)-1,3-DIMETHYL-5,6-DIHYDROTHYMINE  
 $C_{13}H_{16}N_2O_4$  (II)

cis-5-HYDROXY-6-(o-HYDROXYPHENYL)-1,3-DIMETHYL-5,6-DIHYDROTHYMINE  
 $C_{13}H_{16}N_2O_4$  (III)

J.L. FLIPPEN-ANDERSON, 1982. *Acta Cryst.*, B38, 125-129.

I. Monoclinic,  $P2_1/n$ ,  $a = 11.100$ ,  $b = 11.326$ ,  $c = 11.026$  Å,  $\beta = 96.2^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.061$  for 2240 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 13.056$ ,  $b = 8.317$ ,  $c = 13.478$  Å,  $\beta = 117.4^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.062$  for 2111 reflexions.

III. Monoclinic,  $P2_1/c$ ,  $a = 8.363$ ,  $b = 12.434$ ,  $c = 12.406$  Å,  $\beta = 103.6^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.097$  for 1274 reflexions.

In molecule I (Fig. 1), the thiophenol adduct, the two rings are linked by the S atom and are essentially parallel to one another. The phenylthio moiety is trans with respect to the methyl group on C(5) and gauche with respect to the hydroxyl on C(6). In both II (Fig. 1) and III (Fig. 1) there is direct C-C bonding between C(6) and the aromatic ring of the phenol. In all three compounds the crystal packing is influenced by O-H...O hydrogen bonds.



2,4-BIS(METHOXYCARBONYLAMINO)-6-(1,2,3,6-TETRAHYDRO-1-PYRIDYL)PYRIMIDINE-3-OXIDE  
 $C_{13}H_{17}N_5O_5$  (I)

7-(METHOXYCARBONYLAMINO)-5-[3,6-DIHYDRO-1(2H)-PYRIDYL]-2-OXO-2H-[1,2,4]OXADIAZOLO-  
 [2,3-a]PYRIMIDINE 3-AZABICYCLO[3.2.2]NONANE  
 $C_{20}H_{28}N_6O_4$  (II)

J.-C. MULLER, H. RAMUZ, J. DALY and P. SCHONHOLZER, 1982. *Helv. Chim. Acta*, **65**, 1454-1466.

I. Triclinic,  $P\bar{1}$ ,  $a = 8.019$ ,  $b = 7.537$ ,  $c = 13.285$  Å,  $\alpha = 84.48$ ,  $\beta = 73.99$ ,  $\gamma = 71.36^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.076$  for 2088 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 12.269$ ,  $b = 9.817$ ,  $c = 9.688$  Å,  $\alpha = 63.07$ ,  $\beta = 95.45$ ,  $\gamma = 93.99^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.054$  for 3324 reflexions.

The analyses established the structures of these two derivatives to be as shown in Fig. 1. In II, the ions are linked by an N-H...N hydrogen bond (2.87 Å).

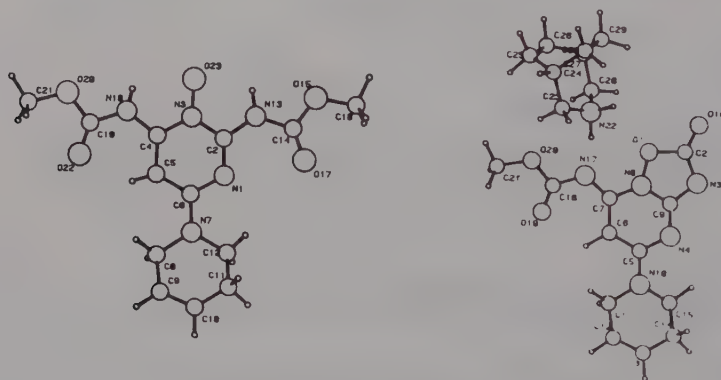


Fig. 1. Views of  $C_{13}H_{17}N_5O_5$  (left) and of  $C_{20}H_{28}N_6O_4$  (right).

3-(ADENIN-9-YL)PROPIONAMIDE 1-METHYLTHYMINE DIHYDRATE  
 $C_{14}H_{18}N_8O_3 \cdot 2H_2O$

$C_8H_{10}N_6O : C_6H_8N_2O_2 \cdot 2H_2O$

M. TAKIMOTO, A. TAKENAKA and Y. SASADA, 1982. *Bull. Chem. Soc. Jpn.*, **55**, 2734-2738.

Monoclinic,  $P2_1$ ,  $a = 11.368$ ,  $b = 6.909$ ,  $c = 12.708$  Å,  $\beta = 114.49^\circ$ ,  $D_m = 1.40$   $Z = 2$   
 Cu radiation,  $R = 0.091$  for 2694 reflexions.

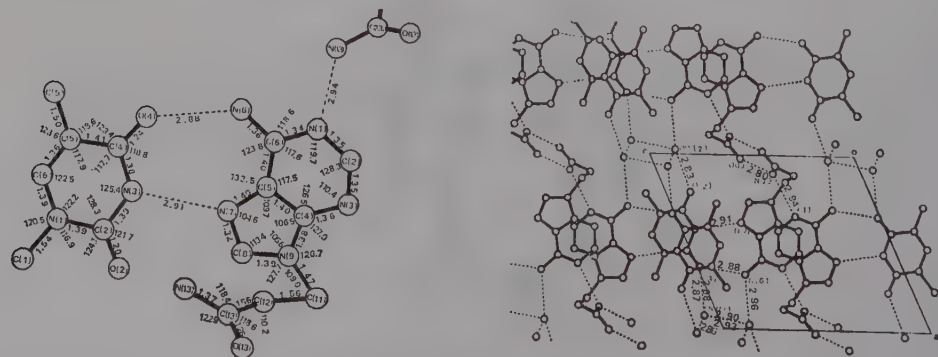
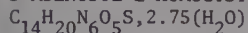


Fig. 1. Dimensions and crystal structure of  $C_8H_{10}N_6O : C_6H_8N_2O_2 \cdot 2H_2O$ .

The base-pairing between adenine and thymine is of Hoogsteen type. The carbamoyl group is hydrogen bonded (Fig. 1) to N(A1) of adenine in this base pair (N...N 2.94 Å). The adenine molecules are linked into sheets perpendicular to the c-axis and base-paired thymine molecules extend from this sheet. Diffuse spots along  $c^*$  can be interpreted as a stacking disorder of the sheets.

S-ADENOSYL-L-HOMOCYSTEINE HYDRATE (form II)



T. ISHIDA, A. TANAKA, M. INOUE, T. FUJIWARA and K. TOMITA, 1982. J. Am. Chem. Soc., 104, 7239-7248.

Monoclinic, C2,  $a = 45.942$ ,  $b = 5.687$ ,  $c = 15.627$  Å,  $\beta = 100.28^\circ$ ,  $D_m = 1.440$ ,  $Z = 8$ . Cu radiation,  $R = 0.12$  for 3243 reflexions.

The conformation of two crystallographically independent molecules (Fig. 1) is anti for the glycosyl bond, C(3')-endo- or C(1')-exo-C(2')-endo sugar puckering, gauche/gauche for the orientation about the C(4')-C(5') bond, trans or gauche<sup>+</sup> about the C( $\alpha$ )-C( $\beta$ ) bond, trans about the C( $\beta$ )-C( $\gamma$ ) bond, and gauche<sup>-</sup> or gauche<sup>+</sup> about the C( $\gamma$ )-S bond.

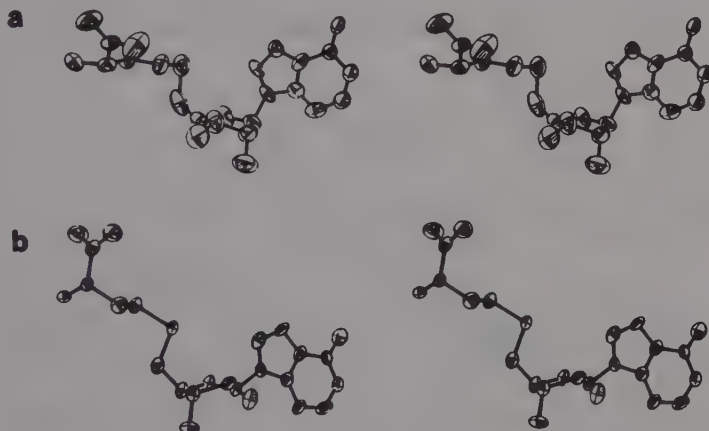
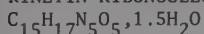


Fig. 1. Stereoviews of the two independent molecules of S-adenosyl-L-homocysteine.

KINETIN RIBONUCLEOSIDE SESQUIHYDRATE



R. WALKER and P. TOLLIN, 1982. Cryst. Struct. Comm., 11, 399-402.

Monoclinic, C2,  $a = 25.35$ ,  $b = 4.680$ ,  $c = 14.31$  Å,  $\beta = 91.80^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.063$  for 585 reflexions.

The structure of this derivative (Fig. 1) is very similar to the parent kinetin (1). The ribose ring adopts the C3' endo-conformation and the C5'-O5' bond is in the gauche-gauche conformation with respect to the ring. The ribose ring adopts the anti-conformation with respect to the purine plane.



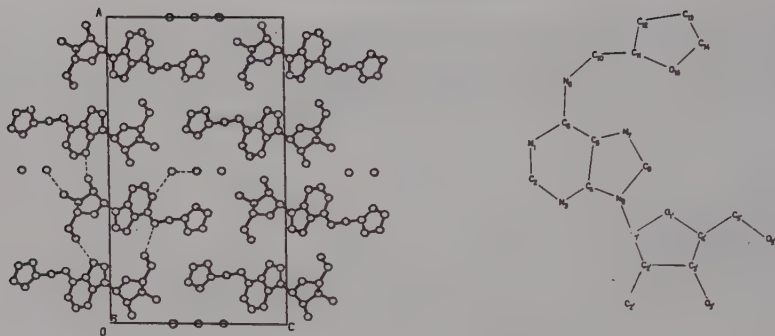
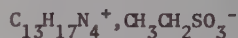


Fig. 1. The  $C_{15}H_{17}N_5O_5$  molecule and a packing diagram.

1. Structure Reports, 43B, 520.

2,4-DIAMINO-5-(1-ADAMANTYL)-6-METHYLPYRIMIDINE ETHANESULPHONATE  
 $C_{15}H_{22}N_4O_3S$  (I)



2,4-DIAMINO-5-(1-NAPHTHYL)-6-METHYLPYRIMIDINE METHANOL SOLVATE  
 $C_{15}H_{14}N_4, CH_4O$  (II)



V. CODY and S.F. ZAKRZEWSKI, 1982. J. Med. Chem., 25, 427-430.

I. Triclinic,  $P\bar{1}$ ,  $a = 10.309$ ,  $b = 14.536$ ,  $c = 6.486$  Å,  $\alpha = 91.76^\circ$ ,  $\beta = 94.98^\circ$ ,  $\gamma = 109.75^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.054$  for 3387 reflexions.

II. Monoclinic,  $C2/c$ ,  $a = 18.542$ ,  $b = 10.990$ ,  $c = 14.721$  Å,  $\beta = 91.905^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.080$  for 1484 reflexions.

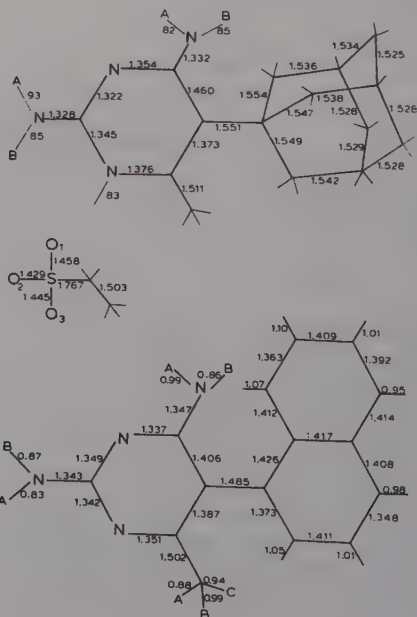
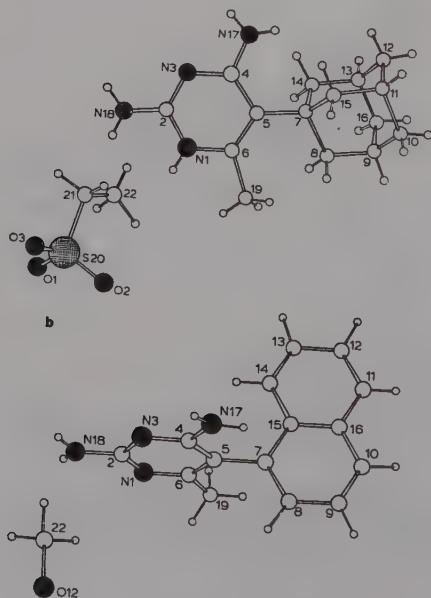


Fig. 1. Molecular structures and bond lengths for  $C_{15}H_{22}N_4O_3S$  (I) (top) and  $C_{15}H_{14}N_4$  (II) (bottom).

The conformation of I is such that the C(7)-C(8) bond is nearly coplanar with the pyrimidine ring (C(8)-C(7)-C(5)-C(6)  $-7.5^\circ$ ) and as a result there is steric interference of the 6-methyl H atoms with the adamantyl ring (Fig. 1). In order to relieve the interference the pyrimidine ring and its substituents are severely distorted from coplanarity. None of these distortions is evident in molecule II. The naphthalene ring is perpendicular to the pyrimidine ring (C(8)-C(7)-C(5)-C(6)  $-87.1^\circ$ ) which is itself planar.

**rac-METHYL 2-HYDROXY-2-(2-THIAMIN)ETHYLPHOSPHONATE CHLORIDE TRIHYDRATE**  
 $C_{15}H_{24}ClN_4O_5PS \cdot 3H_2O$

A. TURANO, W. FUREY, J. PLETCHER, M. SAX, D. PIKE and R. KLUGER, 1982. J. Am. Chem. Soc., **104**, 3089-3095.

Monoclinic,  $P2_1/c$ ,  $a = 9.916$ ,  $b = 16.840$ ,  $c = 15.786$  Å,  $\beta = 119.45^\circ$ ,  $D_m = 1.402$ ,  $Z = 4$ . Mo radiation,  $R = 0.067$  for 1587 reflexions.

The thiamin portion of the molecule assumes the S conformation that is characteristic of other C(2)-substituted thiamins. However, in contrast to previously studied derivatives the conformation of the phosphalactyl substituent is unique in that its OH group is in close contact with the bridging methylene instead of the thiazolium ring S atom and the bond to the phosphonate is oriented perpendicular to the ring plane. The structure of the cation is shown in Fig. 1.

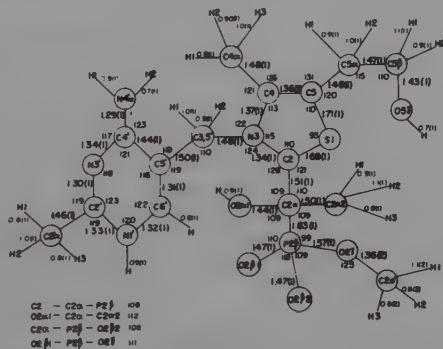


Fig. 1. Bond lengths and angles in the  $(C_{15}H_{24}N_4O_5PS)^+$  cation.

**CYTOSINIUM OCTAMOLYBDATE TETRAHYDRATE**

$C_{16}H_{12}Mo_8N_{12}O_{30} \cdot 4(H_2O)$

$(\text{cyt} \cdot H^+)_4 [Mo_8O_{26}]^{4-} \cdot 4(H_2O)$

A. HOUNTAS and S.E. FILIPPAKIS, 1982. Cryst. Struct. Comm., **11**, 1087-1090.

Triclinic,  $P\bar{1}$ ,  $a = 10.144$ ,  $b = 10.422$ ,  $c = 11.649$  Å,  $\alpha = 103.04^\circ$ ,  $\beta = 109.02^\circ$ ,  $\gamma = 105.18^\circ$ ,  $Z = 1$ . Mo radiation,  $R = 0.042$  for 1748 reflexions.

Fig. 1 represents the molecular arrangement of the two cytosine rings in the asymmetric unit which are hydrogen-bonded between N1(A)...O(B) and N1(B)...O(A). The planes of the cytosinium rings A and B are approximately parallel forming an angle of  $6.1^\circ$ . There are in the unit cell two pairs of doubly hydrogen-bonded cytosine rings. The octamolybdate ion  $(Mo_8O_{26})^{4-}$  consisting of eight  $MoO_6$  octahedra centred by the Mo atoms is sandwiched between the two pairs of the cytosine rings. The minimum and maximum interatomic distances between Mo-O and O...O within the octahedra are 1.67-2.50 and 2.55-3.07 Å and the distances Mo-Mo are 3.21-3.56 Å.

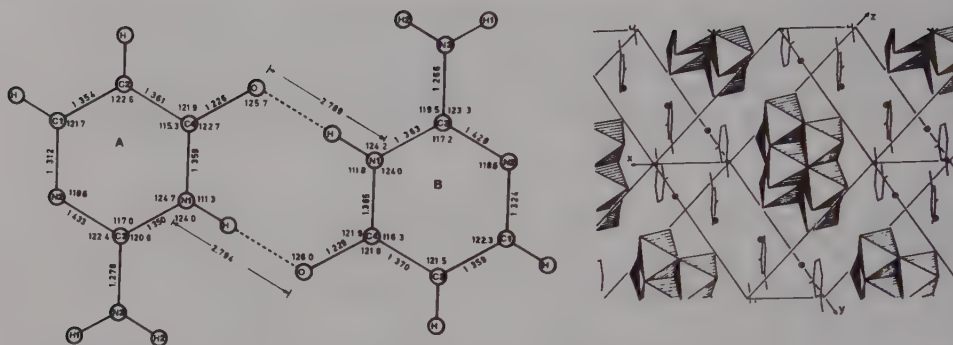


Fig. 1. The cytosinium ion and molecular packing in cytosinium octamolybdate tetrahydrate.

# 9-[3-(3-INDOLYL)PROPYL]ADENINE

$C_{16}H_{16}N_6$

G. BUNICK and D. VOET, 1982. *Acta Cryst.*, **B38**, 575-580.

Monoclinic,  $P2_1/n$ ,  $a = 14.751$ ,  $b = 8.239$ ,  $c = 12.160$  Å,  $\beta = 97.94^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.073$  for 1469 reflexions.

The adenine residues form infinite chains linked by pairs of hydrogen bonds (Fig. 1). While the indole and adenine groups of the same molecule are out of contact with one another the indole group is hydrogen bonded to adenine of a neighbouring molecule by a distorted  $N(1)-H(1) \cdots N(7)$  hydrogen bond, with  $N(7)$  acting as acceptor for two hydrogen bonds. The r.m.s. deviation of the nine atoms of the purine group and those of the indole moiety from their least squares planes is 0.012 and 0.016 Å respectively. The angle between these planes is  $83.5^\circ$ . There are no stacking associations between the rings. Bond lengths and angles are normal.

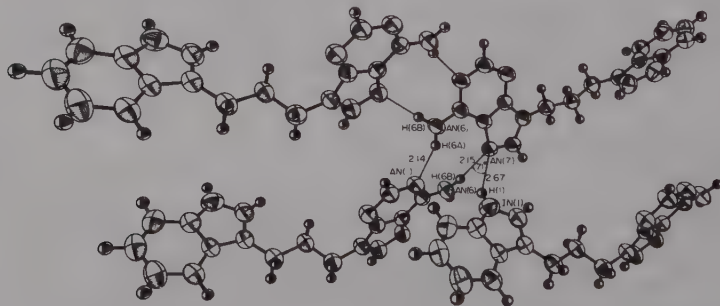


Fig. 1. Intermolecular associations in the 9-[3-(3-indolyl)propyl]adenine structure.

# 1,8-DI(1-THYMINYL)-3,6-DIOXAOCANE

$C_{16}H_{22}N_4O_6$

J.-P. BIDEAU, C. COURSEILLE and G. BRAVIC, 1982. *Acta Cryst.*, **B38**, 2075-2077.

Triclinic,  $P\bar{1}$ ,  $a = 12.356$ ,  $b = 9.341$ ,  $c = 8.524$  Å,  $\alpha = 116.08^\circ$ ,  $\beta = 96.10^\circ$ ,  $\gamma = 92.58^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.041$  for 2704 reflexions.

The triethylenedioxy chain (Fig. 1) does not appreciably disturb the stacking capacity of the thymine groups which are arranged almost parallel and overlapping with separations of 3.19 and 3.41 Å. Bond distances and angles in both rings are very similar and comparable with those in related molecules. One ring has a boat and the other a chair conformation. Equivalent rings in adjacent molecules are linked by pairs of N-H...O bonds (2.934(3) and 2.893(3) Å) across centres of symmetry. There are short CH...O contacts with d(HO) 2.20(3) Å.

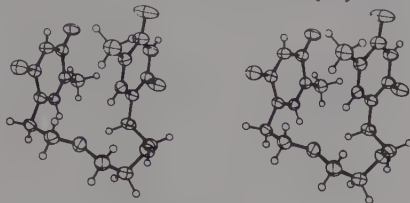


Fig. 1. Stereoview of the  $C_{16}H_{22}N_4O_6$  molecule.

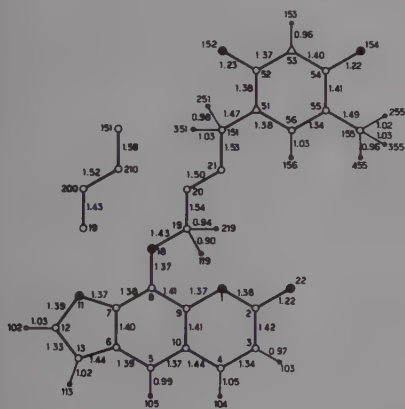
# 9-(1-THYMINYLBUTOXY)PSORALEN

$C_{20}H_{18}N_2O_6$

C. COURSEILLE, G. BRAVIC, J.-P. BIDEAU, J. LHOMME and J.L. DECOUT, 1982. *Acta Cryst.*, B38, 1252-1255.

Monoclinic, Cc,  $a = 8.646$ ,  $b = 25.692$ ,  $c = 8.599$  Å,  $\beta = 113.81^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.04$  for 965 reflexions.

In the molecule (Fig. 1) the polymethylene bridge, linking the psoralen moiety to the pyrimidine base, shows two statistical positions for C(20) and C(21). In the crystal there are intermolecular hydrogen bonds between thymine rings and psoralen rings, but no stacking is present.



The crystal structure consists of monomers (Fig. 1) hydrogen bonded between O(2) and HO(6) and between O(5) and HO(2), with O(2)...O(6) 2.688 Å and O(5)...O(2) 2.850 Å. The internal contact O(2)...O(1) is 2.715 Å and this coupled with the angle O(2)-HO(2)...O(1) of 108° suggests a bifurcated hydrogen bond. The carboxylic acid grouping (O(1), O(6), C(1) and C(2)) is planar but the condensed carbonate-ring system is not. The best plane through O(3), O(4), C(5) and O(5) has C(3) and C(4) 0.04 and 0.13 Å beneath it. Selected bond distances are: C(5)-O(5) 1.207, C(5)-O(4) 1.327, C(5)-O(3) 1.341, C(4)-O(4) 1.448, C(3)-O(3) 1.447 and C(3)-C(4) 1.531 Å.

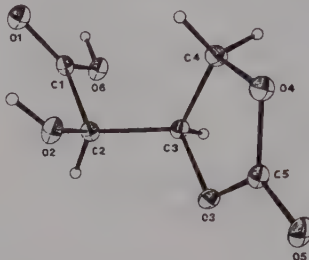


Fig. 1. Molecular structure of  $C_5H_6O_6$ .

#### 1,6:2,5-DIANHYDRO- $\alpha$ -L-GULOFURANOSE



P. KOLL, H.-G. JOHN and J. KOPF, 1982. *Justus Liebigs Ann. Chem.*, 626-638.

Orthorhombic,  $P2_12_12_1$ ,  $a = 5.661$ ,  $b = 9.774$ ,  $c = 21.792$  Å,  $Z = 8$ . Mo radiation,  $R = 0.045$  for 1440 reflexions.

Six-membered rings (1,3- and 1,4- dioxanes) found in the molecule (Fig. 1) adopt almost ideal twist-boat (skew) conformations, while the furanose and oxolane rings show conformations intermediate between T and E. The H atom on O(3) is involved in a bifurcated hydrogen bond to O(2) of the same molecule and O(3) or O(1) of neighbouring molecules (O-H...O 2.681(4)-3.245(4) Å).

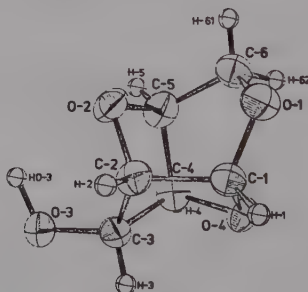


Fig. 1. Perspective view of  $C_6H_8O_4$ .

#### 1-AMINO-1-DEOXY-1,2-N,O-THIOXOMETHYLENE- $\beta$ -D-LYXOPYRANOSE



P. BRIARD, R. ROQUES, G. GOSSELIN, J.L. IMBACH, J.L. MONTERO, J.P. DECLERCQ and G. GERMAIN, 1982. *Acta Cryst.*, B38, 1027-1030.

Orthorhombic,  $P2_12_12_1$ ,  $a = 10.588$ ,  $b = 7.728$ ,  $c = 9.693$  Å,  $Z = 4$ . Cu radiation,  $R = 0.069$  for 651 reflexions.

The molecule (Fig. 1) has a pyranic cyclic structure and a  $\beta$ -[1,2] substitution. The pyran ring is in the chair conformation and the thioxooxazolidine ring is not planar. Intermolecular contacts correspond to van der Waals interactions.

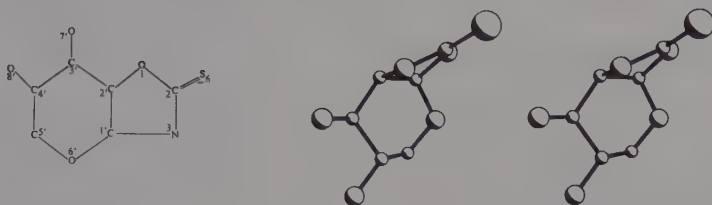
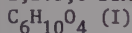
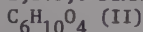


Fig. 1. The  $C_6H_9NO_4S$  molecule and a stereoview.

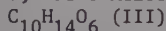
1,2:5,6-DIANHYDROGALACTITOL ( $\alpha$ -form)



1,2:5,6-DIANHYDROGALACTITOL ( $\beta$ -form)



3,4-DI-O-ACETYL-1,2:5,6-DIANHYDROGALACTITOL



M. CZUGLER, K. SIMON, L. INSTITÓRIS, I. VIDRA and I. CSÖREGH, 1982. Carbohydr. Res., 108, 173-180.

I. Monoclinic,  $P2_1/c$ ,  $a = 5.092$ ,  $b = 5.433$ ,  $c = 12.359$  Å,  $\beta = 94.5^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.115$  for 528 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 4.989$ ,  $b = 7.957$ ,  $c = 9.496$  Å,  $\alpha = 113.75^\circ$ ,  $\beta = 98.90^\circ$ ,  $\gamma = 78.90^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.061$  for 784 reflexions.

III. Triclinic,  $P\bar{1}$ ,  $a = 9.442$ ,  $b = 7.997$ ,  $c = 8.380$  Å,  $\alpha = 93.43^\circ$ ,  $\beta = 105.22^\circ$ ,  $\gamma = 106.52^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.062$  for 1805 reflexions.

In all three compounds, the molecules lie about inversion centres (one molecule in I and two independent molecules in II and III). Whereas only one conformer is found in I, (type A) (Fig. 1) two conformers are found in II, (types A and B) (Fig. 1). In III, the two molecules are of type B conformation (Fig. 1). Type B molecules are derived from type A by a rotation of  $\sim 120^\circ$  about C2-C3.



Fig. 1. Views of  $\alpha$ - $C_6H_{10}O_4$  (left), the two  $\beta$ - $C_6H_{10}O_4$  molecules (centre) and one of the  $C_{10}H_{14}O_6$  molecules (right).



1,6-ANHYDRO- $\beta$ -D-MANNOPYRANOSE

H. MALUSZYNSKA, Y. KINOSHITA and G.A. JEFFREY, 1982. *Carbohydr. Res.*, **100**, 17-28.

Orthorhombic,  $P2_12_12_1$ ,  $a = 10.971$ ,  $b = 13.935$ ,  $c = 9.012$  Å,  $D_m = 1.565$ ,  $Z = 8$ . Mo radiation,  $R = 0.043$  for 2355 reflexions.

The two symmetry-independent molecules in the unit cell have similar conformations (Fig. 1), except for the orientation of one of the three hydroxyl groups. The conformation of the pyranose rings is  ${}^1C_4$  distorted towards  $E_0$ , and that of the anhydro rings is  ${}^0E$ . There are significant differences between the two molecules in two of the four C-O bond-lengths. These occur where there are important differences in the hydrogen-bonding environment of the oxygen atoms. The molecules are hydrogen-bonded by three linear and three bifurcated O-H...O interactions which form four-membered loops linked into infinite chains.

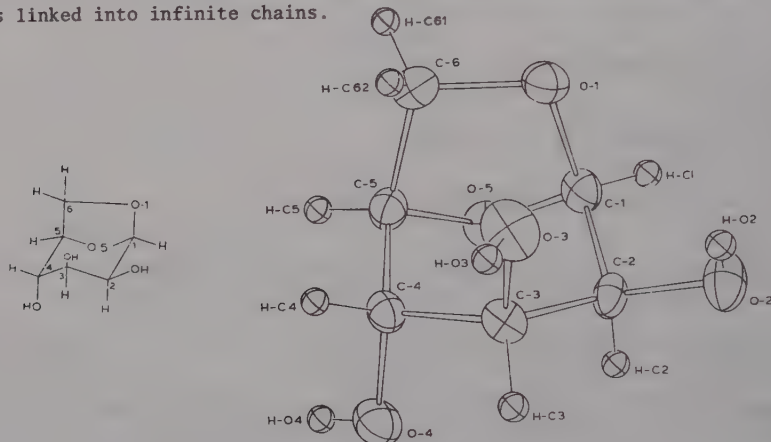


Fig. 1. A view of one of the  $\text{C}_6\text{H}_{10}\text{O}_5$  molecules.

## GALACTARIC ACID (MUCIC ACID)



G.A. JEFFREY and R.A. WOOD, 1982. *Carbohydr. Res.*, **108**, 205-211.

Triclinic,  $P\bar{1}$ ,  $a = 4.900$ ,  $b = 5.728$ ,  $c = 6.784$  Å,  $\alpha = 92.32$ ,  $\beta = 93.74$ ,  $\gamma = 93.08^\circ$ ,  $D_m = 1.790$ ,  $Z = 1$ . Mo radiation,  $R = 0.034$  for 787 reflexions (at  $-147^\circ\text{C}$ ).

The molecule (Fig. 1) lies on an inversion centre. The torsional angles along the straight chain differ slightly from exact linearity, with  $\text{C1-C2-C3-C3}'$   $-174^\circ$ . The carboxylic acid group is almost planar, with  $\text{H(O1A)-O1A-C1-O1B}$   $-1.8^\circ$ . The characteristic, planar conformation of an  $\alpha$ -hydroxy carboxylic acid group is also observed, with  $\text{O1A, O1B, C1, C2, O2}$  in a plane, with maximum deviations of  $0.096$  Å for  $\text{C2}$  and  $0.077$  Å for  $\text{O2}$ . The molecules are linked in the crystal structure by strong hydrogen bonds which involve all of the functional groups, as shown in Fig. 1. The carboxylic acid groups form characteristic, hydrogen-bond dimers across the centres of symmetry, which link the molecules end-to-end in chains. The hydroxyl groups hydrogen-bond into equally characteristic squares, which link the dimerized, molecular chains laterally. There is, in addition, a weak, bifurcated interaction from one hydroxyl,  $\text{O3...H}$ , to the carboxylate oxygen,  $\text{O1B}$ , on an adjacent chain.



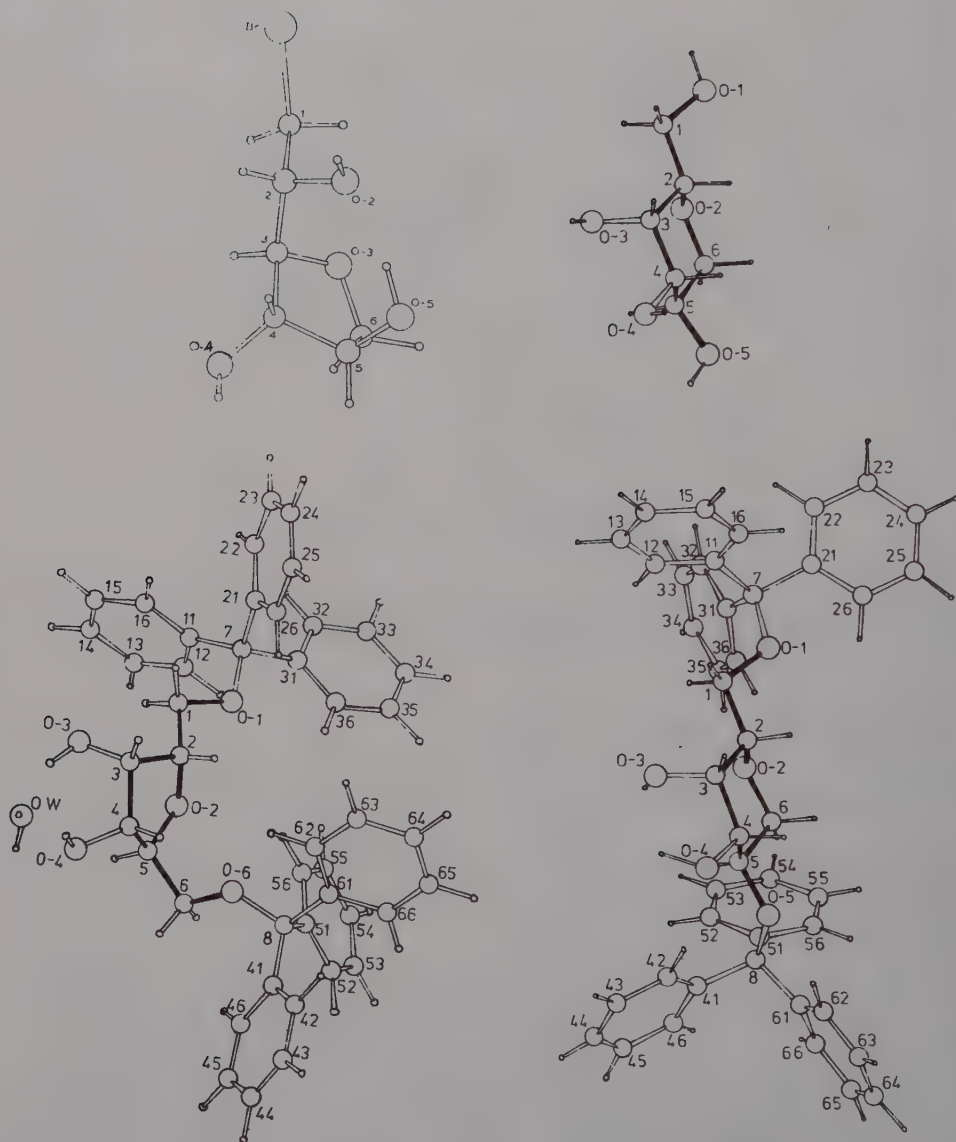


Fig. 1. Views of  $C_6H_{11}BrO_4$  (I) (top left),  $C_6H_{12}O_5$  (II) (top right),  $C_4H_4O_5 \cdot H_2O$  (III) (lower left) and  $C_4H_5O_5$  (IV) (lower right).

# GLUCURONAMIDE HYDRATE

$C_6H_{11}NO_6 \cdot H_2O$

P.R. PERRIER and S.R. BYRN, 1982. *J. Org. Chem.*, **47**, 4677-4680.

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.426$ ,  $b = 18.114$ ,  $c = 6.708$  Å,  $Z = 4$ . Cu radiation,  $R = 0.033$  for 759 reflexions.

Glucuronamide hydrate crystals contain the  $\alpha$  anomer of glucuronamide (Fig. 1). Analysis of the crystal packing of glucuronamide shows that it has both a relatively small water tunnel (area =  $1.05 \text{ \AA}^2$ ) and three relatively strong hydrogen bonds between water and glucuronamide. The results substantiate the correlation between crystal packing (i.e., tunnel area and hydrogen bonding) and the threshold temperature of desolvation.

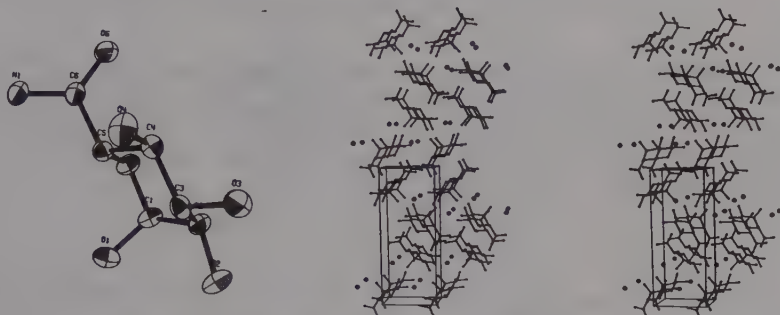


Fig. 1. A glucuronamide molecule and a stereoview of the crystal packing of the hydrate.

4 $\beta$ -D-RIBOFURANOSYL-1,2,4-TRIAZOLIN-3-ONE

$C_7H_{11}N_3O_5$  (I)

2 $\beta$ -D-RIBOFURANOSYL-1,2,4-TRIAZOLIN-3-ONE

$C_7H_{11}N_3O_5$  (II)

D.R. HAINES, N.J. LEONARD and D.F. WIEMER, 1982. J. Org. Chem., 47, 474-482.

I. Orthorhombic,  $P2_12_12_1$ ,  $a = 5.726$ ,  $b = 9.294$ ,  $c = 16.583 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.054$  for 984 reflexions.

II. Orthorhombic,  $P2_12_12_1$ ,  $a = 7.073$ ,  $b = 7.373$ ,  $c = 17.475 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.057$  for 780 reflexions.

These two triazolinone monoriboside isomers were differentiated by  $^{13}\text{C}$  NMR long-range coupling patterns and the assignments confirmed by X-ray analysis. Both the position of N-ribosidation and the configurations at C1' (Fig. 1) were determined for both isomers. In I and II, the atoms of the triazoline rings are coplanar and the bond lengths are short, consistent with some aromatic character. The furanose rings have C2'-endo conformations. There is extensive intermolecular hydrogen bonding in both crystals. In crystals of II, there is stacking of the triazolinone moieties.

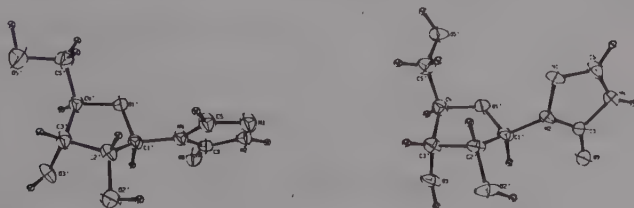


Fig. 1. Views of isomer I (left) and II (right) of  $C_7H_{11}N_3O_5$ .

METHYL  $\alpha$ -L-IDOSEPTANOSIDE

C.T. GRAINGER, S. RUKVICHAI and J.D. STEVENS, 1982. *Cryst. Struct. Comm.*, **11**, 1939-1944.

Trigonal,  $P3_1$ ,  $a = 10.529$ ,  $c = 6.757 \text{ \AA}$ ,  $Z = 3$ . Cu radiation,  $R = 0.024$  for 963 reflexions.

The seven-membered ring (Fig. 1) adopts the twist-chair conformation  $^{5,6}\text{TC}_{3,4}$  in which the pseudo axis of symmetry passes through C1. This conformation corresponds to the low energy conformation, twist-chair C, of the parent heterocycle, oxepan. The molecules are linked by hydrogen bonds as shown in Fig. 1. There are two unsymmetrical bifurcated interactions each of which incorporates a weak intra-molecular hydrogen bond.

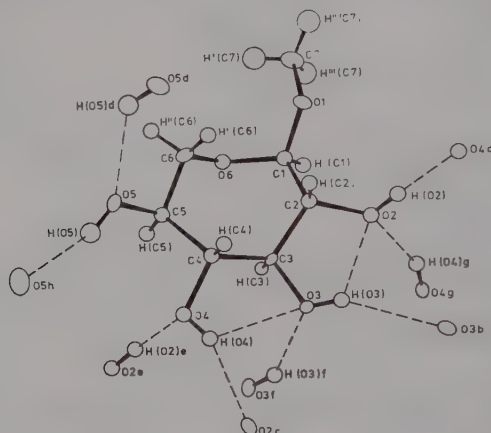


Fig. 1. The  $\text{C}_7\text{H}_{14}\text{O}_6$  molecule and hydrogen bonding.

2,3,5-O-ORTHOACETYL-1,6-ANHYDRO- $\alpha$ -L-GULOFURANOSE

P. KOLL, H.-G. JOHN and J. KOPF, 1982. *Justus Liebigs Ann. Chem.*, 639-650.

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.404$ ,  $b = 9.935$ ,  $c = 10.736 \text{ \AA}$ ,  $Z = 4$ . Mo radiation,  $R = 0.039$  for 1172 reflexions.

The structure was established as that shown in Fig. 1. Bond lengths and angles have normal values.

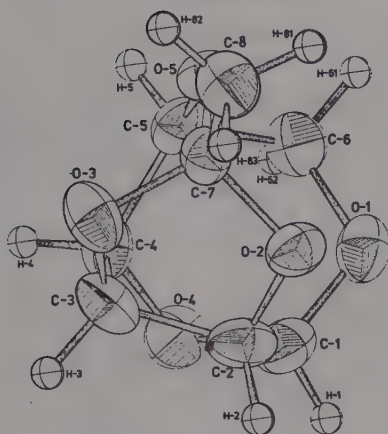


Fig. 1. The structure of  $C_8H_{10}O_5$ .

1,2-O-(2,2,2-TRICHLOROETHYLIDENE)- $\alpha$ -D-GLUCOFURANOSE ( $\alpha$ -D-CHLORALOSE)  
 $C_8H_{11}Cl_3O_6$

T. TAGA, T. KAJI and K. OSAKI, 1982. *Acta Cryst.*, B38, 1874-1876.

Hexagonal,  $P3_2$ ,  $a = 13.383$ ,  $c = 5.692$  Å,  $D_m = 1.755$ ,  $Z = 3$ . Mo radiation,  $R = 0.05$  for 992 reflexions.

The rigidly-fused glucofuranose and dioxolane rings (Fig. 1) have  $^3T_4$  (pseudo-rotation phase angle  $P$   $35.5^\circ$ ) and  $O(2)_E$  conformations respectively. Molecules are packed in a left-handed helix along  $c$ . The furanose-ring oxygens form intermolecular hydrogen-bonds with  $O(3)$  hydroxyl groups. Intermolecular hydrogen bonds between  $O(5)$  and  $O(6)$  hydroxyl groups form right-handed double-helix chains around a threefold screw axis. There are short  $Cl \dots Cl$  contacts ( $3.45$  Å) about another threefold screw axis.

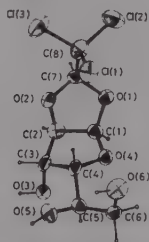


Fig. 1. The  $\alpha$ -D-chloralose molecule.

METHYL 7-DEOXY-D-glycero- $\beta$ -D-galacto-HEPTOPYRANOSIDE MONOHYDRATE  
 $C_8H_{16}O_6 \cdot H_2O$  (I)

METHYL 7-DEOXY-L-glycero- $\beta$ -D-galacto-HEPTOPYRANOSIDE  
 $C_8H_{16}O_6$  (II)

D. AVENEL, J. OHANESSIAN, H. GILLIER-PANDRAUD and P. BOULLANGER, 1982. *Acta Cryst.*, B38, 225-231.

I. Monoclinic,  $C2$ ,  $a = 15.63$ ,  $b = 4.711$ ,  $c = 16.24$  Å,  $\beta = 109.09^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.038$  for 1101 reflexions.



II. Orthorhombic,  $P2_12_12_1$ ,  $a = 25.05$ ,  $b = 7.758$ ,  $c = 5.083 \text{ \AA}$ ,  $Z = 4$ . Mo radiation,  $R = 0.077$  for 861 reflexions.

In I (Fig. 1) the hydroxyl group  $O(6)H$  and the methyl group  $C(7)H_3$  are in the gauche-trans and trans-gauche conformations, respectively, relative to  $O(4)$  and  $O(5)$ ; in II (Fig. 1) they are in the reverse positions. In both crystals molecules are associated by hydrogen bonds in 'hydrophilic' regions clearly separated from 'hydrophobic' regions.

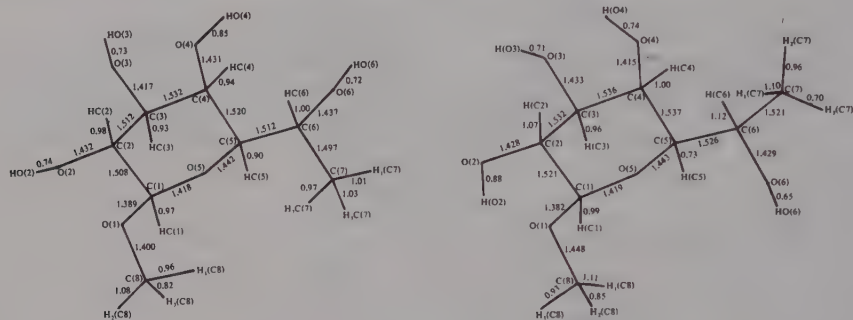


Fig. 1. Bond distances in  $C_8H_{16}O_6 \cdot H_2O$  (I) (left) and in  $C_8H_{16}O_6$  (II) (right).

# METHYL (METHYL 3-DEOXY- $\alpha$ -D-ARABINO-2-HEPTULOPYRANOSID)ONATE $C_9H_{16}O_7$

D. CHARON, L. SZABO, M. CESARIO and J. GUILHEM, 1982. J. Chem. Soc. Perkin I, 3055-3063.

Orthorhombic,  $P2_12_12_1$ ,  $a = 14.409$ ,  $b = 12.302$ ,  $c = 6.547 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.029$  for 1002 reflexions.

The molecule (Fig. 1) is in a conventional  $^5C_2$  chair conformation, the conformational angles around the ring ranging from  $50.15$  to  $59.8$  compared with a range of  $55.8$  to  $61.7^\circ$  for an ideal pyranose ring. The ring is most puckered at  $O(6)$  and least at  $C(5)$ , and atoms  $C(2)$ ,  $C(3)$ ,  $C(5)$  and  $C(6)$  are nearly coplanar. C-O bond distances range from  $1.408$  to  $1.442 \text{ \AA}$  the  $C(2)-O(6)$  value of  $1.408 \text{ \AA}$  being significantly shorter than the average, possibly due to the coplanarity of  $O(6)$ ,  $C(2)$ ,  $C(1)$  and  $O(10)$ . The carbon bond angles in the interior of the ring vary from  $110.6$  to  $111.8^\circ$  and the exterior angles range from  $106.4$  to  $114.2^\circ$ . The molecular packing is determined by a set of intramolecular helicoid hydrogen bonds. The spirals of strong hydrogen bonds are extended in the direction  $c$  and involve the ring  $O(5)H$  and the side chain  $O(7)H$ .

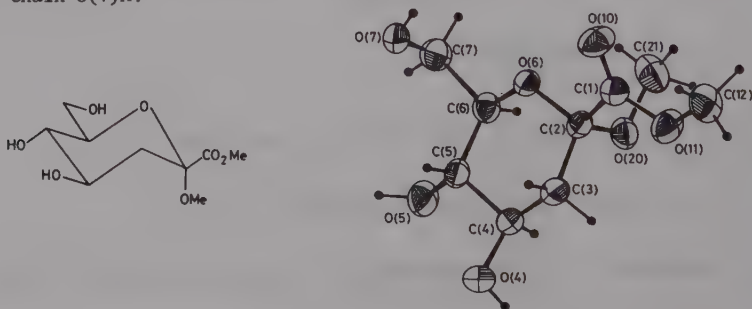
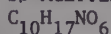


Fig. 1. Molecular skeleton and structure of  $C_9H_{16}O_7$ .

1 $\beta$ -ACETYLRUBRIANITROSE

H. HOEKSEMA, S.A. MIZSAK, L. BACZYNSKYJ and L.M. PSCHIGODA, 1982. J. Am. Chem. Soc., 104, 5173-5181.

Monoclinic,  $P2_1$ ,  $a = 8.528$ ,  $b = 13.133$ ,  $c = 11.741$  Å,  $\beta = 112.38^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.033$  for 2286 reflexions (at  $-155^\circ\text{C}$ ).

The molecule is shown in Fig. 1. No unusual geometrical features were noted.

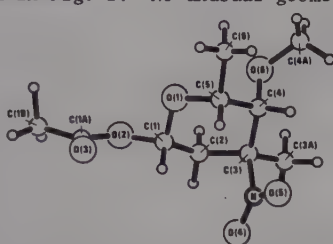


Fig. 1. The 1 $\beta$ -acetylrubrianitroso molecule.

1-N-ACETYL- $\beta$ -D-GLUCOPYRANOSYL-AMINE

C.A. BUSH, K. BLUMBERG and J.N. BROWN, 1982. Biopolymers, 21, 1871-1977.

Orthorhombic,  $P2_12_12_1$ ,  $a = 9.002$ ,  $b = 15.318$ ,  $c = 8.851$  Å,  $Z = 4$ . Mo radiation,  $R = 0.07$  for 858 reflexions.

The pyranose ring (Fig. 1) in the crystal is in the  ${}^4\text{C}_1$  chair conformation and the amide functions at C1 and C2 are both oriented such that the amide protons are nearly trans to their respective sugar-ring protons. Coupling constants determined from the fully assigned proton NMR spectrum in aqueous solution are consistent with the conformation in the crystal.

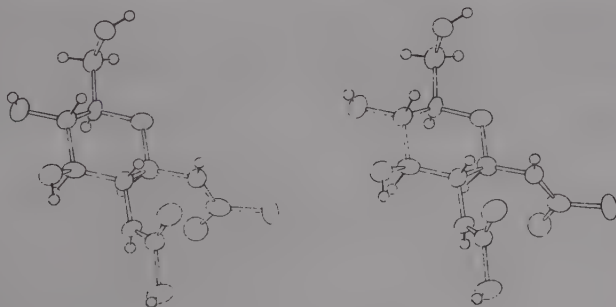


Fig. 1. A stereoview of  $\text{C}_{10}\text{H}_{18}\text{N}_2\text{O}_6$ .

2,3,4-TRI-O-ACETYL- $\alpha$ -D-XYLOPYRANOSYLFLUORIDE

P. LUGER, J. BUSCHMANN, H.-J. SCHMIDT and H. PAULSEN, 1982. Acta Cryst., B38, 2732-2735.

Orthorhombic,  $P2_12_12_1$ ,  $a = 15.429$ ,  $b = 10.581$ ,  $c = 7.939$  Å (at 115 K);  $a = 15.840$ ,  $b = 10.716$ ,  $c = 8.037$  Å (at 295 K),  $Z = 4$ . Mo radiation,  $R = 0.029$  for 1637 reflexions (at 115 K).

In the molecule (Fig. 1) the pyranosyl ring has a regular  ${}^4C_1$  chair form. The bond lengths around the anomeric C atom are in accordance with the consequences of the anomeric effect. The axial C-F bond length (1.397(2) Å) agrees well with previous ab initio calculations. No unusually short intermolecular contacts are observed.

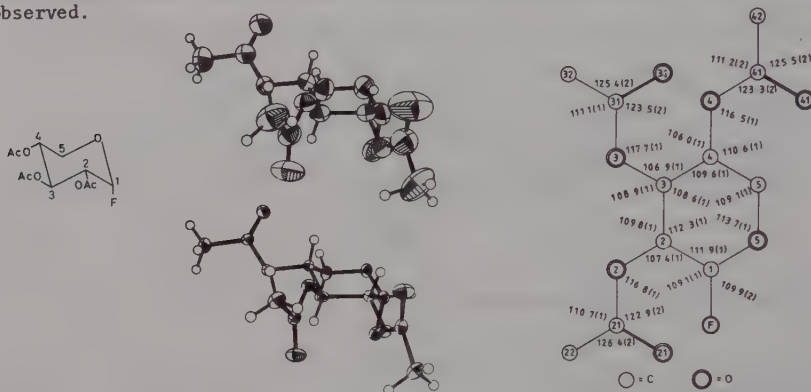


Fig. 1.  $C_{11}H_{15}FO_7$ : perspective views of the molecule at 295 K (upper left) and at 115 K (lower left) and bond angles at 115 K (right).

### 3,4-DINITROPHENYL- $\beta$ -D-GLUCOPYRANOSIDE HYDRATE

$C_{12}H_{14}N_2O_{10} \cdot x(H_2O)$  ( $1.5 < x < 2$ )

P.G. JONES, A.J. KIRBY, R. GLENN and E. HADICKE, 1982. *Z. Krist.*, **161**, 127-136.

Monoclinic,  $P2_1$ ,  $a = 14.267$ ,  $b = 7.486$ ,  $c = 15.368$  Å,  $\beta = 101.10^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.038$  for 3477 reflexions.

The two independent molecules show some torsion angle differences about the C(1)-O(1) bond (Fig. 1) but are otherwise similar. The acetal C-O bond lengths are C(1)-O(1) 1.405 and 1.410(4), C(1)-O(10) 1.405 and 1.397(5) Å. A partially occupied water site leads to a disorder in the hydrogen bonding network in the crystal.

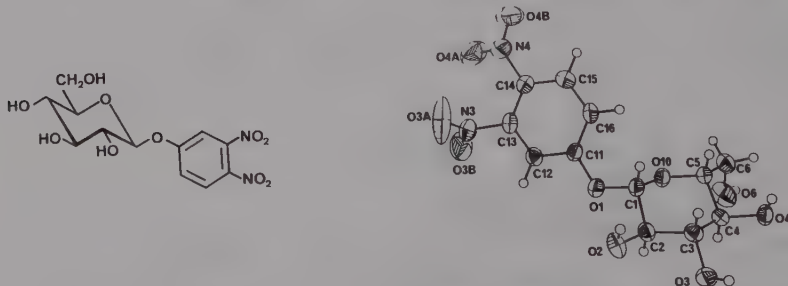


Fig. 1. The  $C_{12}H_{14}N_2O_{10}$  molecule.

### 2-NITROPHENYL- $\beta$ -D-GLUCOPYRANOSIDE

$C_{12}H_{15}NO_8$

P.G. JONES, A.J. KIRBY, R. GLENN and E. HADICKE, 1982. *Z. Krist.*, **161**, 79-85.

Orthorhombic,  $P2_12_12_1$ ,  $a = 4.700$ ,  $b = 13.314$ ,  $c = 20.876$  Å,  $Z = 4$ . Cu radiation,  $R = 0.031$  for 1486 reflexions.

The acetal bond lengths in the molecule (Fig. 1) are  $C(1)-O(1)$  1.409(4) and  $C(1)-O(10)$  1.411(3) Å and are not significantly different. The structure is stabilised by a system of  $O-H...O$  hydrogen bonds.

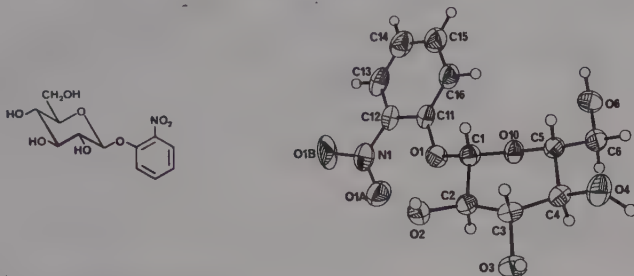


Fig. 1. The  $C_{12}H_{15}NO_8$  molecule.

4-NITROPHENYL- $\alpha$ -D-GLUCOPYRANOSIDE  
 $C_{12}H_{15}NO_8$

P. SWAMINATHAN, 1982. *Acta Cryst.*, B38, 184-188.

Monoclinic,  $P2_1$ ,  $a = 28.810$ ,  $b = 6.747$ ,  $c = 6.729$  Å,  $\beta = 103.68^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.039$  for 1900 reflexions. [See also the following Report.]

In the two independent molecules (Fig. 1) the valence angles at the bridge oxygen atom  $O(1')$  ( $117.3^\circ$  and  $120.4^\circ$ ) are significantly different. In both molecules the endocyclic C-O bond lengths are unequal, with  $C(1')-O(5')$  and  $C(5')-O(5')$ , respectively, 1.408 and 1.452 Å (molecule A) and 1.411 and 1.446 Å (molecule B), whilst the glycosidic bond lengths are close to the mean C-O bond length. The molecules are linked in the crystal structure by hydrogen bonds.

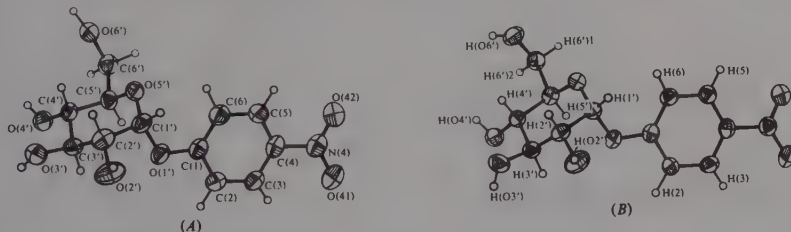


Fig. 1.  $C_{12}H_{15}NO_8$ : perspective views of molecules A and B.

4-NITROPHENYL- $\alpha$ -D-GLUCOPYRANOSIDE  
 $C_{12}H_{15}NO_8$

P.G. JONES, G.M. SHELDRICK, A.J. KIRBY and W.B.T. CRUSE, 1982. *Z. Krist.*, 161, 69-77.

Monoclinic,  $P2_1$ ,  $a = 28.045$ ,  $b = 6.767$ ,  $c = 6.719$  Å,  $\beta = 90.30^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.032$  for 2462 reflexions. [See also the preceding Report.]

The two independent molecules differ mainly in relative ring orientation. One molecule is shown in Fig. 1. The bond lengths at the acetal centre are not significantly different ( $C(1)-O(1)$  1.415 and 1.420(3),  $C(1)-O(10)$  1.415 and 1.414(4) Å). In the crystal, the two independent molecules occupy separate domains

in the lattice. A series of hydrogen bonds connects the glucose rings of both molecules.

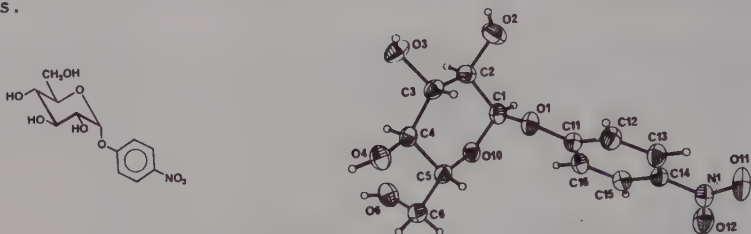


Fig. 1. A  $C_{12}H_{15}NO_8$  molecule.

# PHENYL- $\alpha$ -D-GLUCOPYRANOSIDE



P.G. JONES, G.M. SHELDRICK, A.J. KIRBY and R. GLENN, 1982. *Z. Krist.*, **160**, 259-267.

Monoclinic,  $P2_1$ ,  $a = 13.108$ ,  $b = 5.138$ ,  $c = 18.392$  Å,  $\beta = 103.23^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.049$  for 3447 reflexions.

The two independent molecules are very similar except for differing torsion angles (and thus ring orientations) about  $C(1)-O(1)$ , and the differing orientations of  $C(6)-O(6)$  (Fig. 1). Bond lengths at the acetal centre are  $C(1)-O(1)$  1.419 and 1.411(5),  $C(1)-O(10)$  1.407 and 1.408(4) Å.

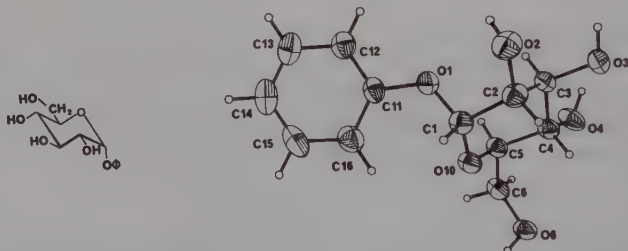
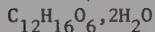


Fig. 1. A  $C_{12}H_{16}O_6$  molecule ( $\alpha$ -isomer).

# PHENYL- $\beta$ -D-GLUCOPYRANOSIDE DIHYDRATE



P.G. JONES, G.M. SHELDRICK, W. CLEGG, A.J. KIRBY and R. GLENN, 1982. *Z. Krist.*, **160**, 269-274.

Orthorhombic,  $P2_12_12_1$ ,  $a = 4.893$ ,  $b = 9.747$ ,  $c = 29.015$  Å,  $Z = 4$ . Mo radiation,  $R = 0.061$  for 1531 reflexions.

The bond lengths at the acetal centre (Fig. 1) are appreciably different (exocyclic  $C(1)-O(1)$  1.394, endocyclic  $C(1)-O(10)$  1.434 Å). The water of crystallization is loosely held and there is probably disorder in the hydrogen bond network.

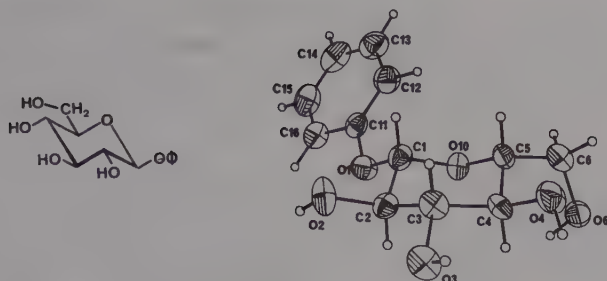


Fig. 1. The  $C_{12}H_{16}O_6$  molecule,  $\beta$ -isomer.

1-BROMO-1-DESOXY-2,3:5,6-DI-O-ISOPROPYLIDENE-1-NITRO- $\alpha$ -D-MANNOFURANOSYL  
 $C_{12}H_{18}BrNO_7$

B. AEBISCHER, A. VASELLA and H.-P. WEBER, 1982. *Helv. Chim. Acta*, **65**, 621-634.

Hexagonal,  $P6_1$ ,  $a = 15.021$ ,  $c = 14.194$  Å,  $Z = 6$ . Cu radiation,  $R = 0.058$  for 1368 reflexions.

Bond lengths have normal values. The mannofuranose ring (Fig. 1) has  $^{\circ}E$  conformation, with the nitro group pseudo-equatorial and the bromine atom pseudo-axial.

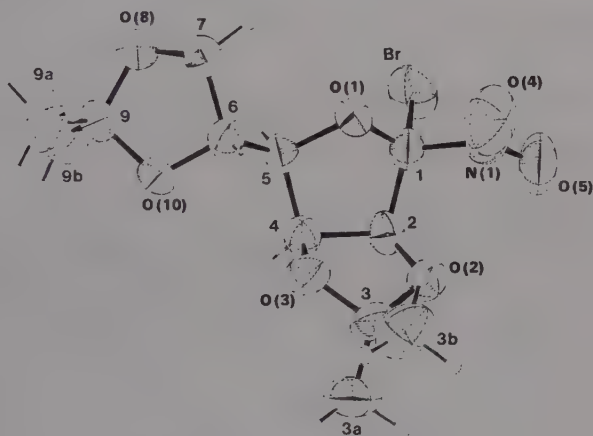


Fig. 1. A view of  $C_{12}H_8BrNO_7$ .

DI-D-FRUCTOSE ANHYDRIDE III  
 $C_{12}H_{20}O_{10}$

T. TANIGUCHI and T. UCHIYAMA, 1982. *Carbohydr. Res.*, **107**, 255-262.

Orthorhombic,  $P2_12_12_1$ ,  $a = 16.438$ ,  $b = 10.483$ ,  $c = 8.0928$  Å,  $D_m = 1.548$ ,  $Z = 4$ . Mo radiation,  $R = 0.063$  for 2034 reflexions.

As shown in Fig. 1, D-fructoses 1 and 2 are respectively the  $\alpha$  and  $\beta$  anomer of D-fructofuranose. The conformations of the moieties in the molecule are as expected, with two puckered furanose rings and a 1,4-dioxane ring fused onto the furanose ring of D-fructose 2. The 1,4-dioxane ring also connects with the ring of D-fructose 1



at the anomeric carbon atom (C2) in a spiro arrangement. The ring of D-fructose 1 has the  ${}^4T_3$  conformation, with C4 displaced by 0.469 Å on the endo side of the plane through atoms C2, C5, and O2, and with C3 displaced by 0.186 Å on the opposite side. The D-fructose 2 moiety has the  ${}^4T_5$  conformation. The C5' and C4' atoms are displaced by 0.338 and 0.064 Å respectively on the opposite sides of the plane through the atoms C2', C3' and O2'. The 1,4-dioxane ring has a skew conformation.

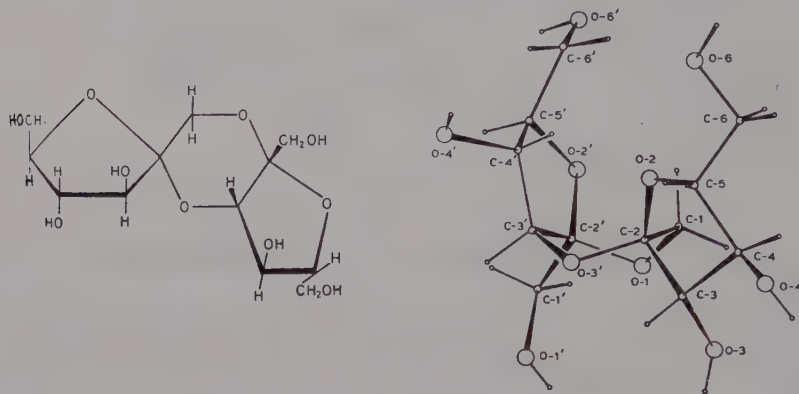
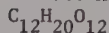


Fig. 1. The di-D-fructose anhydride III structure.

D-threo-HEXO-2,5-DIULOSE DIMER



L.K. HANSEN, A. HORDVIK and R. HOVE, 1982. *Acta Chem. Scand.*, **B36**, 301-309.

Orthorhombic,  $P2_12_12_1$ ,  $a = 12.094$ ,  $b = 21.700$ ,  $c = 5.329$  Å,  $D_m = 1.68$ ,  $Z = 4$ . Mo radiation,  $R = 0.04$  for 1443 reflexions.

The molecule contains a pyranose part, numbered 1-6 and a furanose part, numbered 11-16 and a dioxolane part (C(5), O(5), C(12), C(13) and O(13)) which occurs as a result of dimerization (Fig. 1). The dioxolane and furanosyl rings have envelope conformations. Average values for C-C, C-O(hydroxyl) and C-O(ether) bond lengths are 1.525, 1.419 and 1.422 Å respectively. There are three intramolecular hydrogen bonds: O(2)...O(1) 2.757, O(15)...O(4) 2.708 and O(16)...O(14) 2.724 Å and five intermolecular hydrogen bonds with O...O distances ranging from 2.741 to 2.942 Å.

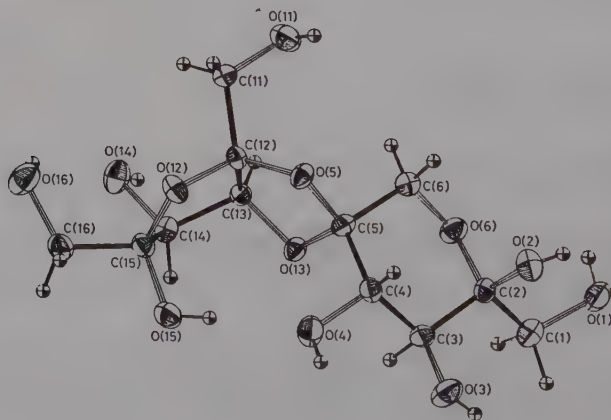


Fig. 1. Molecular structure of  $C_{12}H_{20}O_{12}$ .

MALTITOL (4-O- $\alpha$ -D-GLUCOPYRANOSYL-D-GLUCITOL) $C_{12}H_{24}O_{11}$ S. OHNO, M. HIRAO and M. KIDO, 1982. Carbohydr. Res., 108, 163-171.Orthorhombic,  $P2_12_12_1$ ,  $a = 8.166$ ,  $b = 12.721$ ,  $c = 13.629$  Å,  $D_m = 1.602$ ,  $Z = 4$ . Mo radiation,  $R = 0.030$  for 1446 reflexions.

The molecule (Fig. 1) shows a fully extended conformation with no intramolecular hydrogen bonds. All nine hydroxyl groups are involved in intermolecular hydrogen-bond networks and in bifurcated, finite chains. The D-glucopyranosyl moiety has the  ${}^4C_1$  conformation, and the conformation about the C5-C6 bond is gauche-gauche. The D-glucitol residue has the bent [ap,Psc,Psc(APP)] conformation.

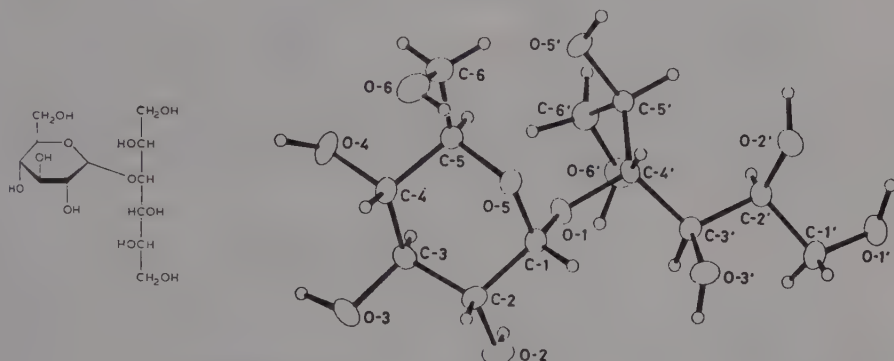
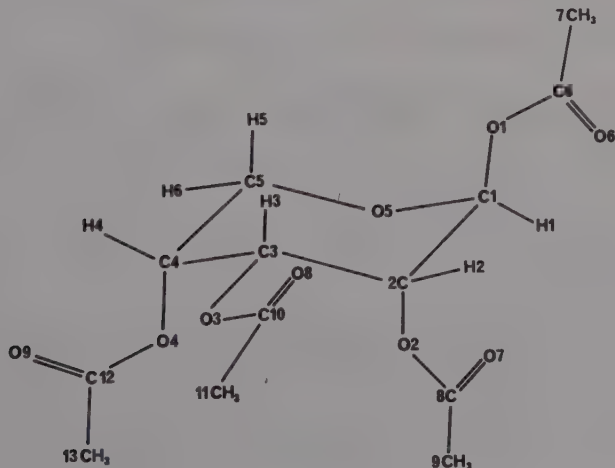
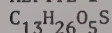


Fig. 1. A view of maltitol.

1,2,3,4-TETRA-O-ACETYL- $\beta$ -DL-RIBOPYRANOSE $C_{13}H_{18}O_9$ V.J. JAMES and J.D. STEVENS, 1982. Cryst. Struct. Comm., 11, 457-461.Monoclinic,  $C2/c$ ,  $a = 11.525$ ,  $b = 10.953$ ,  $c = 25.258$  Å,  $\beta = 93.00^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.048$  for 2546 reflexions.Fig. 1 The  $C_{13}H_{18}O_9$  structure.

The molecule (Fig. 1) adopts the chair conformation,  ${}^1C_4$ , in which the acetoxyl group at C1 is axially oriented. The ring is somewhat flattened in the region of C2. Molecular dimensions are in accord with expected values.

# HEPTYL 1-THIO- $\alpha$ -D-MANNOPYRANOSIDE



D.C. CARTER, J.R. RUBLE and G.A. JEFFREY, 1982. Carbohydr. Res., 102, 59-67.

Orthorhombic,  $P2_12_12_1$ ,  $a = 6.600$ ,  $b = 7.624$ ,  $c = 30.24$  Å,  $D_m = 1.245$ ,  $Z = 4$ . Mo radiation,  $R = 0.030$  for 2320 reflexions (at 123 K).

The six-membered ring (Fig. 1) has the  ${}^4C_1$  conformation. The ring C-O bond lengths are significantly different, C1-O5 1.433(2), C5-O5 1.448(2) Å, but the C-S bond lengths, 1.819(2), 1.824(2) Å, are not. The molecules pack in a bilayer arrangement, with the hexyl chains parallel, and head-to-tail in adjacent molecules. The hydrogen bonding of the pyranoside moieties consists of infinite chains cross-linked through bifurcated bonds to the ring-oxygen atoms. The compound has a liquid-crystal phase lying between 64°C and the melting point at 151-152°C, with a periodicity of 21 Å.

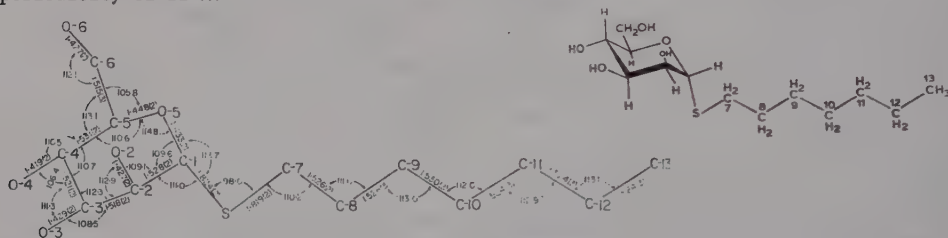
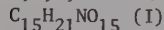


Fig. 1. The  $C_{13}H_{26}O_5S$  molecule.

# 3,4,5,7-TETRA-O-ACETYL-2,6-ANHYDRO-D-GLYCERO-D-IDO-HEPTONAMIDE



# 3,4,5,7-TETRA-O-ACETYL-2,6-ANHYDRO-D-GLYCERO-L-GLUCO-HEPTONAMIDE



M. SUNDARALINGAM, R.G. BRENNAN, P. SWAMINATHAN, T.P. HAROMY, W.B. DRENDEL, J.N. BEMILLER, M. CHMIELEWSKI and D.P. CERRETTI, 1982. J. Carbohydr. Chem., 1, 85-103.

I. Monoclinic,  $P2_1$ ,  $a = 8.864$ ,  $b = 8.346$ ,  $c = 13.569$  Å,  $\beta = 114.12^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.040$  for 1911 reflexions.

II. Monoclinic,  $P2_1$ ,  $a = 15.045$ ,  $b = 8.106$ ,  $c = 7.491$  Å,  $\beta = 97.23^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.046$  for 2122 reflexions.

Both compounds (Fig. 1) adopt the unusual  ${}^2C_5$  ( ${}^1C_4$ ) chair conformation with the carbamoyl groups on the anomeric carbon atoms equatorially oriented. In this conformation the orientations of the substituents are 2e, 3a, 4a, 5a and 6a in I and 2e, 3a, 4a, 5e and 6a in II which leads to unfavourable 1,3-diaxial interactions. The  ${}^2C_5$  conformation is stabilized by the N-H...O intramolecular hydrogen bond between the carbamoyl nitrogen atom and the pyranosyl ring oxygen atom. Semi-empirical energy calculations reveal that the rotational freedom of the carbamoyl group is greater for the equatorial orientation ( ${}^2C_5$ ) than for the axial orientation ( ${}^4C_1$ ).

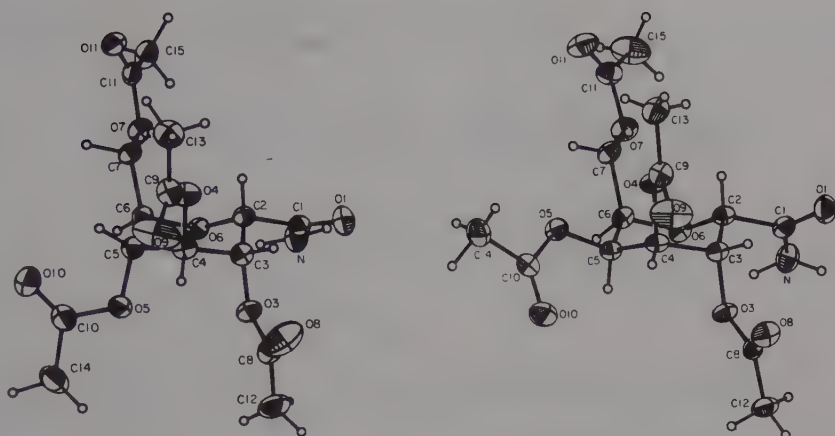


Fig. 1. Views of the D-ido- (left) and D-gluco- (right) isomers of  $C_{15}H_{21}NO_{15}$ .

METHYL 2,3,4,5-TETRA-O-ACETYL- $\beta$ -D-ALLOSEPTANOSIDE

$C_{15}H_{22}O_{10}$

V.J. JAMES and J.D. STEVENS, 1982. Cryst. Struct. Comm., 11, 79-83.

Monoclinic,  $P2_1$ ,  $a = 8.036$ ,  $b = 12.185$ ,  $c = 9.986$  Å,  $\beta = 112.0^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.031$  for 1772 reflexions.

The seven-membered ring has a twist-chair conformation (Fig. 1) with a pseudo axis of symmetry passing through  $C(1)^{5,6}T_{3,4}$ .

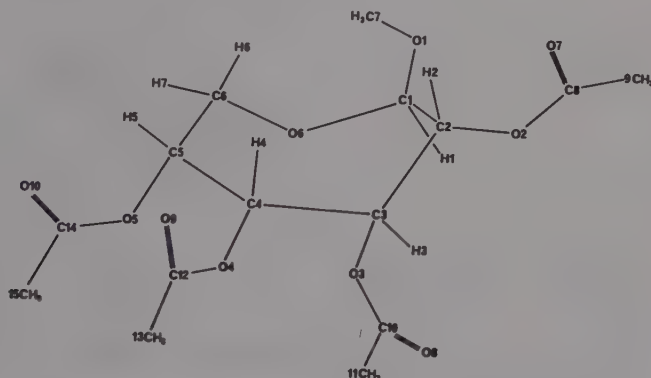


Fig. 1. The conformation of  $C_{15}H_{22}O_{10}$ .

METHYL 2,3,4,5-TETRA-O-ACETYL- $\alpha$ -L-IDOSEPTANOSIDE

$C_{15}H_{22}O_{10}$

V.J. JAMES and J.D. STEVENS, 1982. Cryst. Struct. Comm., 11, 1933-1938.

Orthorhombic,  $P2_12_12_1$ ,  $a = 19.754$ ,  $b = 21.408$ ,  $c = 8.508$  Å,  $Z = 8$ . Cu radiation,  $R = 0.046$  for 2542 reflexions.

The seven-membered ring (Fig. 1) adopts the  $^{5,6}TC_{3,4}$  conformation with the

pseudo symmetry axis passing through C1. This corresponds to the lowest energy conformation, twist-chair C<sub>2</sub>, of the parent heterocycle oxepan.

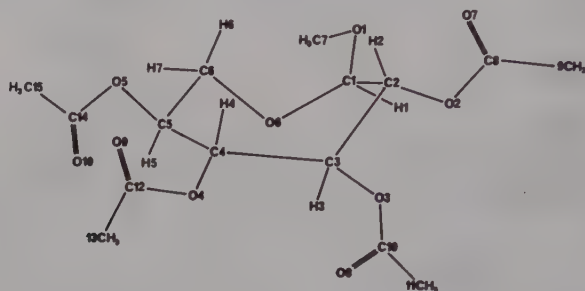


Fig. 1. A view of C<sub>15</sub>H<sub>22</sub>O<sub>10</sub>.

1,2,3,4,6-PENTA-O-ACETYL- $\alpha$ -D-GLUCOPYRANOSE  
C<sub>16</sub>H<sub>22</sub>O<sub>11</sub>

P.G. JONES, G.M. SHELDRICK, A.J. KIRBY and R. GLENN, 1982. *Z. Krist.*, **161**, 237-243.

Orthorhombic, P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 5.592, *b* = 14.724, *c* = 23.966 Å, *Z* = 4. Mo radiation, *R* = 0.046 for 2965 reflexions.

In the molecule (Fig. 1) the trend towards longer exocyclic bonds at the acetal centre in compounds with strongly electronegative aglycones is maintained. The C(1)-O(1) and C(1)-O(10) bond lengths are 1.431(4) and 1.403(4) Å respectively.

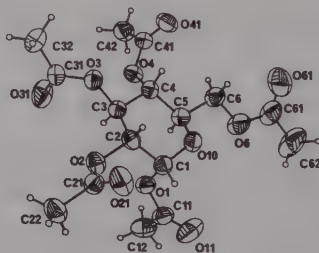
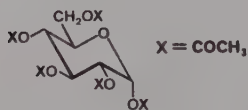


Fig. 1. The  $\alpha$ -form of C<sub>16</sub>H<sub>22</sub>O<sub>11</sub>.

1,2,3,4,6-PENTA-O-ACETYL- $\beta$ -D-GLUCOPYRANOSE  
C<sub>16</sub>H<sub>22</sub>O<sub>11</sub>

P.G. JONES, G.M. SHELDRICK, A.J. KIRBY and R. GLENN, 1982. *Z. Krist.*, **161**, 245-251.

Orthorhombic, P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 5.747, *b* = 14.182, *c* = 24.480 Å, *Z* = 4. Mo radiation, *R* = 0.087 for 2059 reflexions.

The acetyl group at C(6) in the molecule (Fig. 1) is disordered. The endocyclic acetal bond is longer than the exocyclic one (C(1)-O(10) 1.426(7), C(1)-O(1) 1.408(8) Å), the opposite of the pattern observed in the preceding report.





hydrogen bonds (Fig. 1) forming a two-stranded anti-parallel  $\beta$ -pleated sheet structure. The two molecules have the same general backbone conformation with the  $\phi_2$ ,  $\psi_2$  and  $\psi_3$  torsional angles close to  $-90^\circ$ ,  $+120^\circ$  and  $+150^\circ$  respectively, but they show different magnitudes of rotational disorder in the thiophene ring as well as disorder in the pyrrolidine ring. The molecules in these dimer fragments are further hydrogen-bonded to molecules along the a and c axes, forming a pronounced two-dimensional predominantly hydrophobic layer structure. These layers, in which the atoms are almost equally arranged on both sides, are separated by ordinary van der Waals' distances. A close correlation between the molecular conformation in the solid state and the preferential conformation in solution is found. It is concluded that the crystalline structure possesses structural features which may be of relevance in the hormone-receptor interaction process.

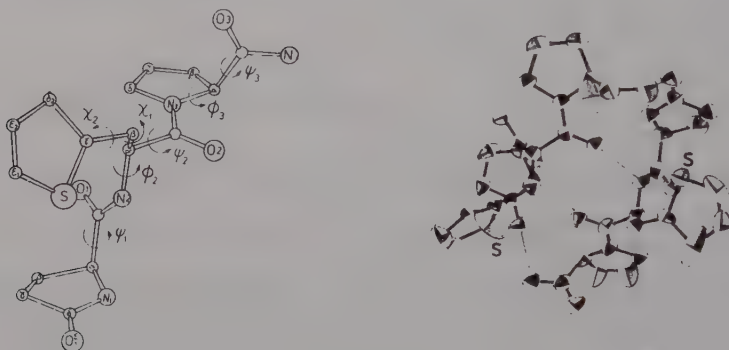


Fig. 1. A  $C_{17}H_{22}N_4O_4S$  molecule and a drawing of a dimer.

1,2:4,5-DIANHYDRO-3-O-(4-PHENYLBENZOYL)XYLITOL  
 $C_{18}H_{16}O_4$

I. VIDRA, L. INSTITORIS, K. SIMON, M. CZUGLER and I. CSOREGH, 1982. Carbohydr. Res., 111, 215-223.

Monoclinic,  $P2_1/c$ ,  $a = 13.709$ ,  $b = 12.467$ ,  $c = 9.144$  Å,  $\beta = 104.06^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.050$  for 1237 reflexions.

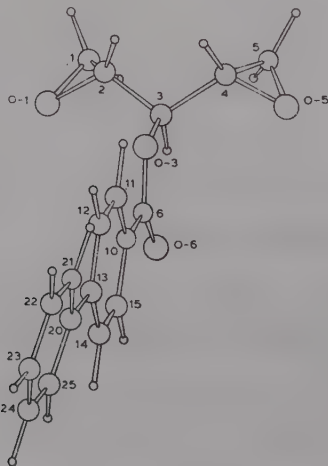


Fig. 1. A view showing the conformation of  $C_{18}H_{16}O_4$ .

The molecule is shown in Fig. 1. The relative positions of the three-membered rings to the O3-C3 bond are  $+sc(85^\circ)$  and  $-sc(-82^\circ)$ . The angle between the best planes of the benzoyl and phenyl groups is  $13^\circ$ .

# OLEFINIC CYCLIC TRIMER OF LÉVOGLUCOSENONE



T.T. STEVENSON, R.E. STENKAMP, L.H. JENSEN, F. SHAFIZADEH and R.E. FURNEAUX, 1982. Carbohydr. Res., 104, 11-19.

Orthorhombic,  $P2_12_12_1$ ,  $a = 18.8695$ ,  $b = 14.1992$ ,  $c = 5.6224 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.033$  for 1291 reflexions.

The hydrogens on the three asymmetric carbon atoms have an all-cis configuration (Fig. 1) projecting from the same side of the molecule as their 1,6-anhydro bridges. This results in a configuration where the pyranoid rings of residues B and C are roughly co-planar and that of residue A lies  $\sim 90^\circ$  to this plane, with the anhydride bridge being exo to the rest of the molecule. The conjugated system of residues B and C is very nearly planar.

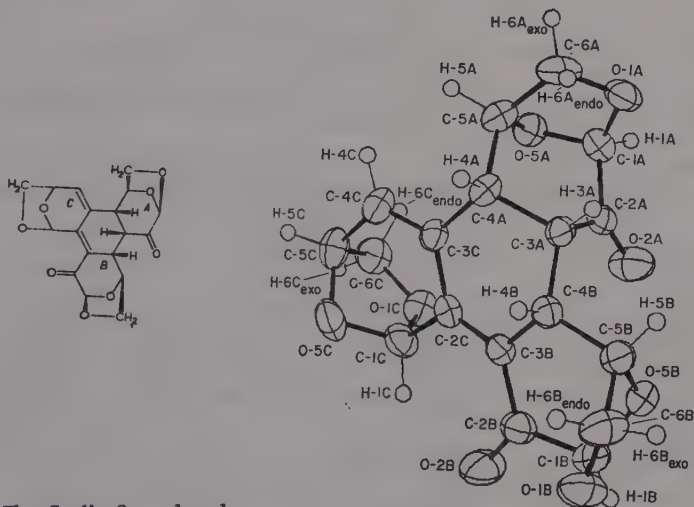
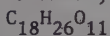


Fig. 1. The  $C_{18}H_{16}O_8$  molecule.

# 2,3,4,6-TETRA-O-ACETYL- $\beta$ -D-MANNOPYRANOSIDE-1-SPIRO-2'-(3'-METHYL-3'-TETRAHYDRO-FURANOL)



G. RÉMY, L. COTTIER, G. DESCOTES, R. FAURE and H. LOISELEUR, 1982. Cryst. Struct. Comm., 11, 235-240.

Monoclinic,  $P2_1$ ,  $a = 8.622$ ,  $b = 15.923$ ,  $c = 8.598 \text{ \AA}$ ,  $\beta = 118.04^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.055$  for 1303 reflexions.

The six-membered ring (Fig. 1) is in chair form and atom O(2) of the five-membered ring is equatorial. Atom C6 is  $0.593 \text{ \AA}$  out of the plane through C(1), C(7), C(8), O(2).

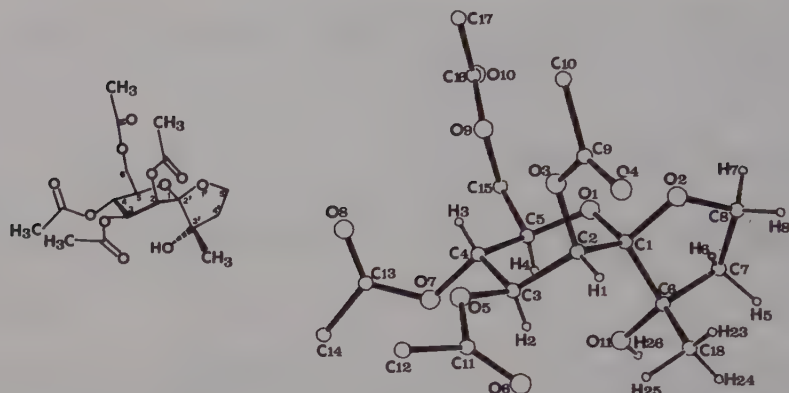
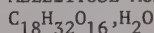


Fig. 1. The  $C_{18}H_{26}O_{11}$  molecule.

MELEZITOSE MONOHYDRATE (form II)



J. BECQUART, A. NEUMAN and H. GILLIER-PANDRAUD, 1982. Carbohydr. Res., 111, 9-21.

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.635$ ,  $b = 15.362$ ,  $c = 19.134$  Å,  $D_m = 1.521$ ,  $Z = 4$ .  
Mo radiation,  $R = 0.045$  for 1971 reflexions.

For details of form I see (1). Bond lengths for both form I and II are in Fig. 1. The glucopyranose rings have  ${}^4C_1$  chair forms. The furanose ring has an  $E_3$  envelope form, with C3' 0.585 Å from the plane of the other four atoms.

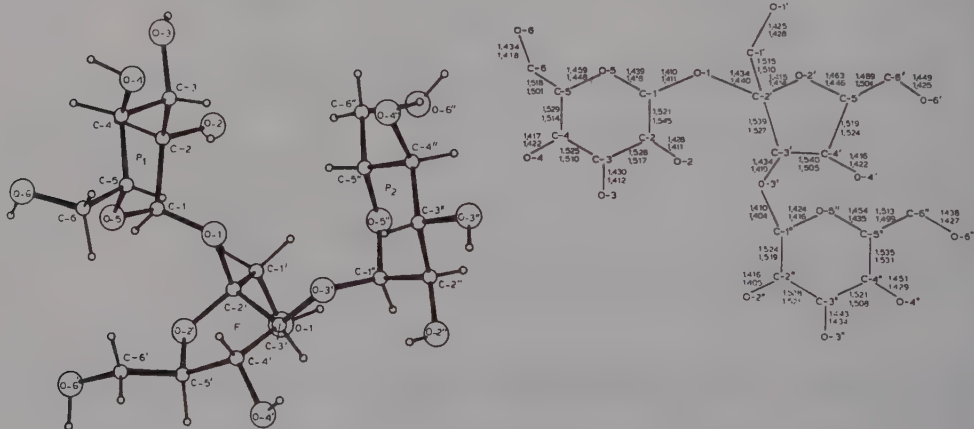


Fig. 1. A view of melezitose from II and bond lengths (form I above form II).

1. Structure Reports, 42B, 334.



Orthorhombic,  $P2_12_12_1$ ,  $a = 5.654$ ,  $b = 16.700$ ,  $c = 25.905$  Å,  $Z = 4$ . Mo radiation,  $R = 0.053$  for 2170 reflexions.

The introduction of four acetyl groups has very little effect on the geometry at the acetal centre C(1) (Fig. 1). The C(1)-O(1) and C(1)-O(10) bond lengths are 1.400(6) and 1.395(6) Å respectively.

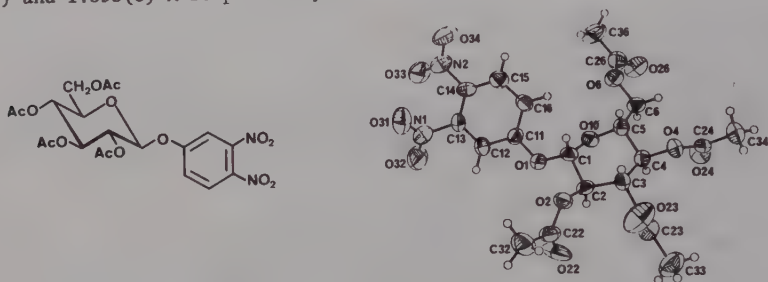


Fig. 1. The  $C_{20}H_{22}N_2O_{14}$  molecule.

1,2,3,4-TETRA-O-ACETYL-5,6-DIDEOXY-5-C-[(S)-PHENYLPHOSPHINYL]-β-L-IDOPYRANOSE  
 $C_{20}H_{25}O_9P$  (I)

1,2,3,4-TETRA-O-ACETYL-5,6-DIDEOXY-5-C-[(S)-PHENYLPHOSPHINYL]-α-L-IDOPYRANOSE  
 $C_{20}H_{25}O_9P$  (II)

S. INOKAWA, K. YAMAMOTO, H. KAWAMOTO, H. YAMAMOTO, M. YAMASHITA and P. LUGER, 1982. Carbohydr. Res., 106, 31-42.

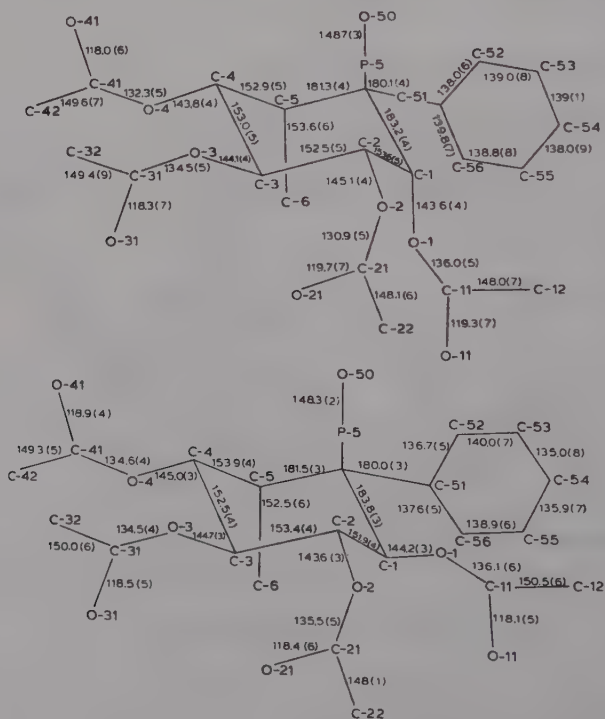


Fig. 1. Bond lengths for the β- (top) and α- (bottom) isomers of  $C_{20}H_{25}O_9P$ .

I. Monoclinic,  $P2_1$ ,  $a = 8.552$ ,  $b = 15.754$ ,  $c = 8.528$  Å,  $\beta = 92.83^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.036$  for 1916 reflexions.

II. Orthorhombic,  $P2_12_12_1$ ,  $a = 16.175$ ,  $b = 13.199$ ,  $c = 10.988$  Å,  $Z = 4$ . Cu radiation,  $R = 0.037$  for 2188 reflexions.

The analyses established the structures shown in Fig. 1. The six-membered rings have chair form and the acetyl groups have the orientation with the C=O bond almost syn-parallel to the corresponding C-H of the ring.

[2(R)-HYDROXY-1(R)-CYCLOHEXYL]-2,3,4,6-TETRA-O-ACETYL- $\beta$ -D-GLUCOPYRANOSIDE  
 $C_{20}H_{30}O_{11}$

J.P. PRALY, G. DESCOTES. R. FAURE and H. LOISELEUR, 1982. Cryst. Struct. Comm., 11, 1323-1327.

Triclinic,  $P1$ ,  $a = 5.628$ ,  $b = 9.488$ ,  $c = 11.117$  Å,  $\alpha = 89.80^\circ$ ,  $\beta = 81.00^\circ$ ,  $\gamma = 78.88^\circ$ ,  $Z = 1$ . Cu radiation,  $R = 0.114$  for 1019 reflexions.

The pyranose ring has chair conformation (Fig. 1) with all side-chains equatorial. There is an intramolecular O(3)-H...O(1) hydrogen bond (O...O 2.79(1) Å).

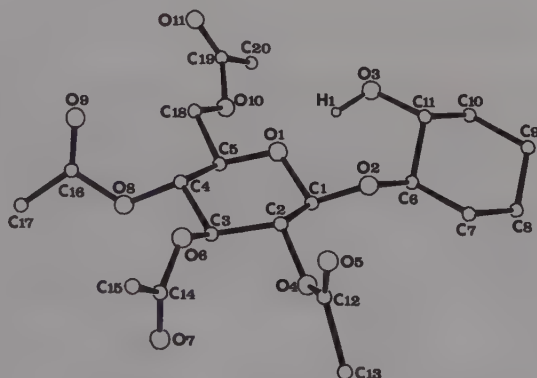


Fig. 1. A view of  $C_{20}H_{30}O_{11}$ .

BENZYL 4-O-(2,3-DIDEOXY- $\alpha$ -L-PENT-2-ENOPYRANOS-4-ULOSYL)-2,3-O-ISOPROPYLIDENE- $\alpha$ -L-RHAMNOPYRANOSIDE

$C_{21}H_{26}O_7$

J.W. KRAJEWSKI, G. GRYNKIEWICZ, P. GLUZINSKI, Z. URBANCZYK-LIPKOWSKA, A. ZAMOJSKI and K. STADNICKA, 1982. Acta Cryst., B38, 1485-1489.

Monoclinic,  $P2_1$ ,  $a = 9.797$ ,  $b = 8.739$ ,  $c = 12.141$  Å,  $\beta = 91.90^\circ$ ,  $D_m = 1.19$ ,  $Z = 2$ . Mo radiation,  $R = 0.043$  for 1281 reflexions.

The unsaturated dihydropyranone ring (A) (Fig. 1) has the  $^oH_5$  half-chair conformation; O(7) and C(11) deviate 0.401 and -0.239 Å from the C(7), C(8), C(9), C(10) plane. The rhamnopyranose ring (B) has the  $^1C_4$  (L) chair conformation strongly deformed towards the twist form due to ring fusion. The methyl group is equatorial. The dioxolane ring (C) has a slightly distorted envelope conformation. The dihedral angles between the mean ring planes are, B-D 77.5(2), B-C 45.3(2), B-A 30.4(3)°. C(7) in A has the S configuration. The interglycosidic  $\phi$  and  $\psi$  angles are 29(4) and 27(2)° respectively.



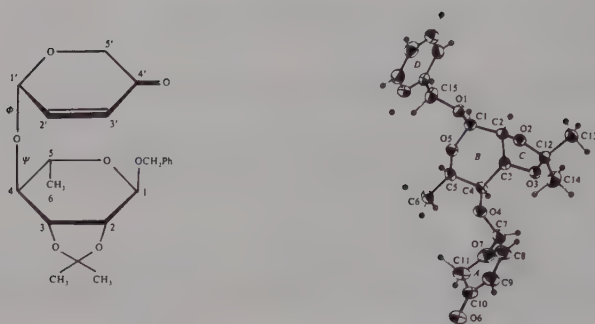


Fig. 1. The  $C_{21}H_{26}O_7$  molecule.

METHYL 4,6-O-BENZYLIDENE-2,3-DIDEOXY-2-[2-(METHOXYCARBONYL)PHENYLAMINO]-3-NITRO- $\beta$ -D-MANNOPYRANOSIDE

$C_{22}H_{24}N_2O_8$

T.E. CONTURO and G.A. JEFFREY, 1982. Carbohydr. Res., 104, 33-40.

Monoclinic,  $P2_1$ ,  $a = 13.267$ ,  $b = 9.633$ ,  $c = 8.839$  Å,  $\beta = 106.26^\circ$ ,  $D_m = 1.311$ ,  $Z = 2$ . Mo radiation,  $R = 0.056$  for 6959 reflexions.

The configuration of the molecule (Fig. 1) was confirmed. The pyranoid conformation is  ${}^4C_1$  with  $Q$  0.611 Å,  $\theta$   $8.5^\circ$ . The dioxolane conformation is close to the ideal chair. The orientation of the anthranilate ring is such that the amine hydrogen atom forms three intramolecular hydrogen-bonds: one major bond to the anthranilate carbonyl oxygen atom, and two minor bonds to the glycosidic oxygen atom and the ring-oxygen atom. The orientation of the anthranilate group is thus fixed such that the angle between the plane of the benzene ring and that of the nitro group is  $116^\circ$ , thereby permitting an intramolecular, C-H...O hydrogen bond between the benzene ortho C-H and the nitro oxygen atom. This bond is normal to the plane of the nitro group, and is believed to be the source of the color phenomenon that prompted this structure analysis.

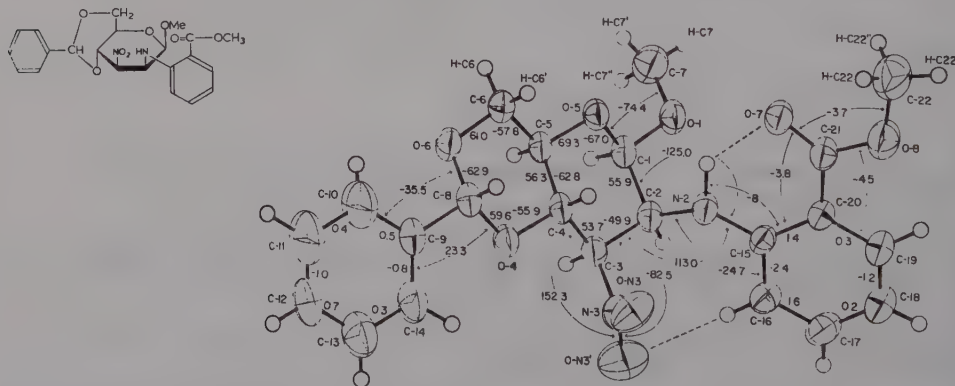


Fig. 1. The  $C_{22}H_{24}N_2O_8$  molecule and principal torsion angles.

2,5-BIS[(1-DEOXY-2,3-ISOPROPYLIDENE- $\alpha$ -D-RIBOFURANOSYL)AMINO]-3,6-DIFLUORO-p-BENZOQUINONE

$C_{22}H_{28}F_2N_2O_{10}$

A. DUBOURG, R. ROQUES, J.L. MONTÉRO, E. GUY, A. MORUZZI, J.L. IMBACH, J.P. DECLERCQ and G. GERMAIN, 1982. *Acta Cryst.*, B38, 1349-1352.

Orthorhombic,  $P2_12_12_1$ ,  $a = 26.500$ ,  $b = 15.544$ ,  $c = 5.615$  Å,  $Z = 4$ . Cu radiation,  $R = 0.051$  for 1665 reflexions.

The molecule (Fig. 1) has  $\alpha$  configuration and the ribofuranose ring has  $E_0$  conformation. The molecules are held together in the crystal by hydrogen bonds.

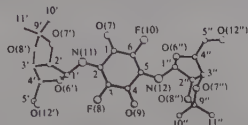
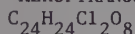


Fig. 1.  $C_{22}H_{28}F_2N_2O_{10}$ : a perspective view of the molecule.

METHYL 4,6-BIS(O-p-CHLOROBENZOYL)-2,3-DIDEOXY-3-C-(METHOXYCARBONYLMETHYL)- $\alpha$ -D-ribo-HEXOPYRANOSIDE



D.L. HUGHES, R.A. PAUPTIT, E. SUBRAMANIAN and J. TROTTER, 1982. *Acta Cryst.*, B38, 2511-2513.

Monoclinic,  $P2_1$ ,  $a = 5.752$ ,  $b = 15.436$ ,  $c = 13.698$  Å,  $\beta = 93.74^\circ$ ,  $D_m = 1.43$ ,  $Z = 2$ . Cu radiation,  $R = 0.042$  for 898 reflexions.

In the molecule (Fig. 1) the six-membered pyranose ring is in the chair conformation, with the bulkier 4,6-p-chlorobenzoyl substituents equatorial, and the 1-OMe and 3-CH<sub>2</sub>CO<sub>2</sub>Me groups axial. Bond lengths and angles and intermolecular distances are normal. The extremities of the p-chlorobenzoyl groups exhibit large thermal vibrations (r.m.s. displacement as large as 0.56(1) Å for one Cl).

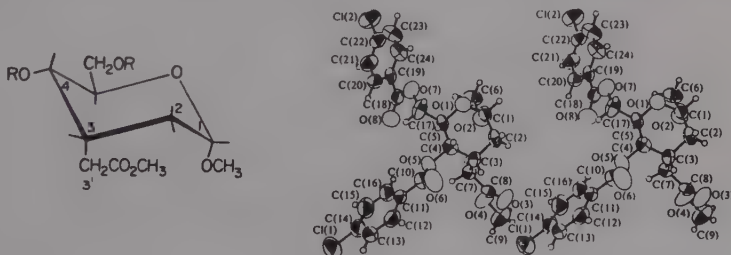
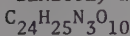


Fig. 1.  $C_{24}H_{24}Cl_2O_8$ : structural formula ( $R = ClC_6H_4CO-$ ) and stereoscopic view of the molecule.

1-DEOXY-2,3-O-ISOPROPYLIDENE-1-[3-(2-METHOXYCARBONYL-PHENYL)UREIDO]-5-O-(p-NITRO-BENZOYL)  $\alpha$ -D-RIBOFURANOSE



R. ROQUES, J.L. MONTÉRO, M. RODRIGUEZ, J.L. IMBACH, J.P. DECLERCQ and G. GERMAIN, 1982. *Acta Cryst.*, B38, 1345-1348.

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.578$ ,  $b = 10.294$ ,  $c = 31.328$  Å,  $Z = 4$ . Mo radiation,  $R = 0.045$  for 1639 reflexions.

The crystal-structure analysis has shown that the molecule (Fig. 1) has  $\alpha$  configuration, that the ribofuranose ring has  $E_0$  conformation, and that the ureido part of the molecule is planar. Bond distances and angles are generally as expected.

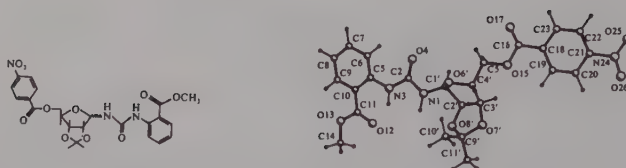


Fig. 1.  $C_{24}H_{25}N_3O_{10}$ : a perspective view of the molecule.

7-DESOXY-1,2:3,4:8,9:11,12-TETRA-O-ISOPROPYLIDENE-7-NITRO- $\beta$ -D-MANNO-D-GLYCERO- $\alpha$ -D-GALACTO-DODECO-1,5-PYRANOSE-7-ULO-7,10-FURANOSE

$C_{24}H_{27}NO_{13}$

B. AEBISCHER, J.H. BIERI, R. PREWO and H. VASELLA, 1982. *Helv. Chim. Acta*, **65**, 2251-2272.

Triclinic,  $P1$ ,  $a = 9.914$ ,  $b = 13.678$ ,  $c = 11.105$  Å,  $\alpha = 67.85^\circ$ ,  $\beta = 74.00^\circ$ ,  $\gamma = 73.99^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.050$  for 8000 reflexions (at  $-140^\circ\text{C}$ ).

The analysis establishes the structure of this dodecosulose (Fig. 1). There are two independent molecules in the unit cell.

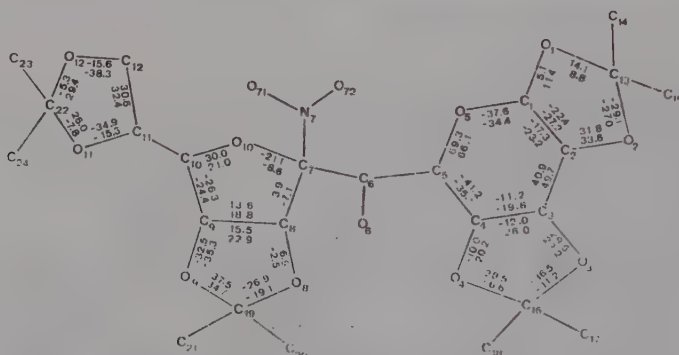


Fig. 1. Torsion angles for the dodecosulose derivative  $C_{24}H_{37}NO_{13}$ .

(Z)-2,3:5,6-DI-O-ISOPROPYLIDENE- $\alpha$ -D-MANNOFURANOSYL-O,N,N-AZOXY-2,3:5,6-DI-O-ISOPROPYLIDENE- $\alpha$ -D-MANNOFURANOSIDE

$C_{24}H_{38}N_2O_{12}$

B.M. AEBISCHER, H.W. HANSEN, A.T. VASELLA and W.B. SCHWEIZER, 1982. *J. Chem. Soc. Perkin I*, 2139-2147.

Monoclinic,  $I2/c$ ,  $a = 19.144$ ,  $b = 5.547$ ,  $c = 25.888$  Å,  $\beta = 88.4^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.037$  for 1755 reflexions.

Bond lengths around the oxy-NNO-azoxy-acetate link of the dimer (Fig. 1) are C(1)-N(1) 1.510, N(1)-O(6) 1.257, N(1)-N(2) 1.283, N(2)-O(7) 1.366 and O(7)-C(21) 1.451 Å.

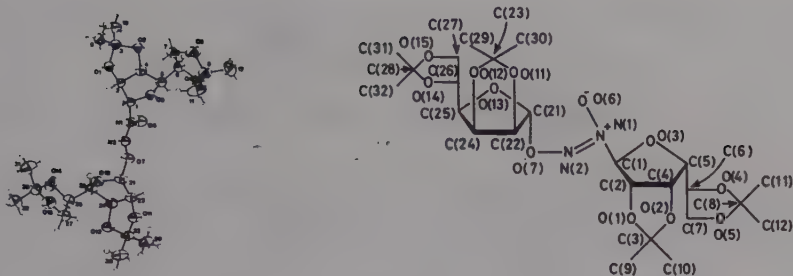


Fig. 1. Molecular skeleton and structure of  $C_{24}H_{38}N_2O_{12}$ .

METHYL 2-(BENZYLOXYCARBONYL)AMINO-2,7-DIDEOXY-3,4:9,10-DI-O-ISOPROPYLIDENE-7-NITRO- $\beta$ -L-GLYCERO-L-ALTRIO-D-GALACTO-UNDECODIALDO-(METHYL-(11R)-FURANOSIDE-(8,11))-PYRANOSIDE-(1,5)

$C_{27}H_{38}N_2O_{13}$

T. SUAMI, Y. FUKUDA, J. YAMAMOTO, Y. SAITO, M. ITO and S. OHBA, 1982. *J. Carbohydr. Chem.*, **1**, 9-19.

Monoclinic,  $P2_1$ ,  $a = 11.197$ ,  $b = 13.117$ ,  $c = 10.399$  Å,  $\beta = 91.58^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.053$  for 3317 reflexions.

The absolute configurations of the chiral centres at C6 and C7 are established (Fig. 1). Bond distances and angles have normal values. Molecules are linked by O(7)-H...O(2) hydrogen bonds (2.719 Å). The molecule is in a maximally extended conformation with an end-to-end distance of 19 Å.

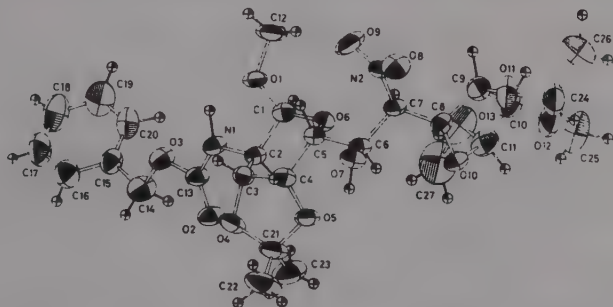


Fig. 1. A view of  $C_{27}H_{38}N_2O_{13}$ .

$\beta$ -MALTOSE OCTAACETATE

$C_{28}H_{38}O_{19}$

F. BRISSE, R.H. MARCHESSAULT, S. PÉREZ and P. ZUGENMAIER, 1982. *J. Am. Chem. Soc.*, **104**, 7470-7476.

Orthorhombic,  $P2_12_12_1$ ,  $a = 5.733$ ,  $b = 23.771$ ,  $c = 25.632$  Å,  $D_m = 1.29$ ,  $Z = 4$ . Cu radiation,  $R = 0.061$  for 3391 reflexions.

The two D-glucose residues have the  ${}^4C_1$  pyranose conformation and are  $\alpha(1\rightarrow4)$  linked. The conformational angles  $\phi$  and  $\psi$  at the glycosidic linkage have the values of  $-29$  and  $-36^\circ$ , respectively. The acetate substituent at C(6) of the reducing residue is in the *gg* conformation, but in the non-reducing residue there is a disorder of the C(6) acetate group. Two distinct orientations are observed in equal

proportions, one having the gt and the other the tg conformation (see Fig. 1).

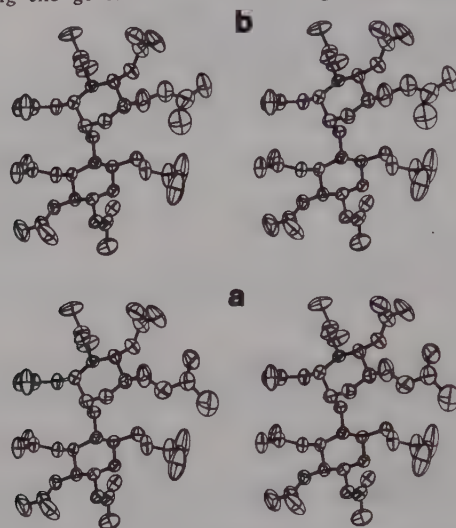
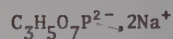
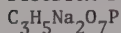


Fig. 1. Stereoviews of  $\beta$ -maltose octaacetate. (a) gt, (b) tg conformation at C(6).

#### DISODIUM D-3-PHOSPHOGLYCERATE



P.F. FEWSTER and R.H. FENN, 1982. *Acta Cryst.*, B38, 282-284.

Monoclinic,  $P2_1$ ,  $a = 9.794$ ,  $b = 5.019$ ,  $c = 7.799$  Å,  $\beta = 111.63^\circ$ ,  $D_m = 2.001$ ,  $Z = 2$ .  
Mo radiation,  $R = 0.063$  for 1146 reflexions.

In the crystal structure (Fig. 1) two intermolecular hydrogen bonds occur and one of these H atoms is also involved in a weak intramolecular hydrogen bond. Bond lengths and angles are generally as expected.

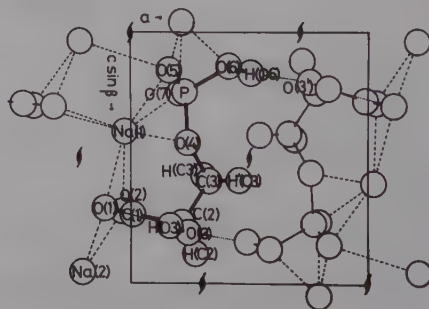
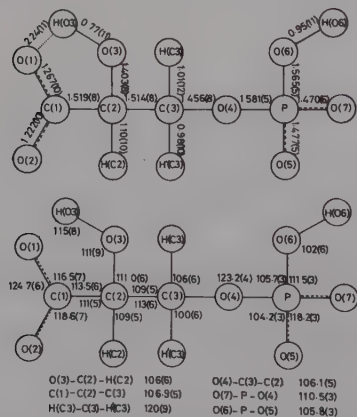
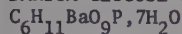


Fig. 1. Bond lengths and angles in the  $\text{C}_3\text{H}_5\text{O}_7\text{P}^{2-}$  anion and a projection of the crystal structure along the b axis.

## BARIUM GLUCOSE 6-PHOSPHATE HEPTAHYDRATE



S.K. KATTI, T.P. SESHADRI and M.A. VISWAMITRA, 1982. Acta Cryst., B38, 1136-1140.

Monoclinic,  $P2_1$ ,  $a = 11.881$ ,  $b = 8.616$ ,  $c = 8.350 \text{ \AA}$ ,  $\beta = 102.95^\circ$ ,  $D_m = 2.09$ ,  $Z = 2$ . Mo radiation,  $R = 0.068$  for 1603 reflexions.

In the anion (Fig. 1) the conformation about the exocyclic C(5)-C(6) bond is gauche-gauche. The phosphate ester bond, P-O(6), is  $1.61 \text{ \AA}$ . The  $\text{Ba}^{2+}$  ion is surrounded by nine O atoms within a distance of  $2.95 \text{ \AA}$ , of which seven are from water molecules (Fig. 1). There is an intramolecular hydrogen bond between the sugar hydroxyl O(4) and phosphate oxygen O(12) with  $\text{O} \cdots \text{O} \ 2.62 \text{ \AA}$ .

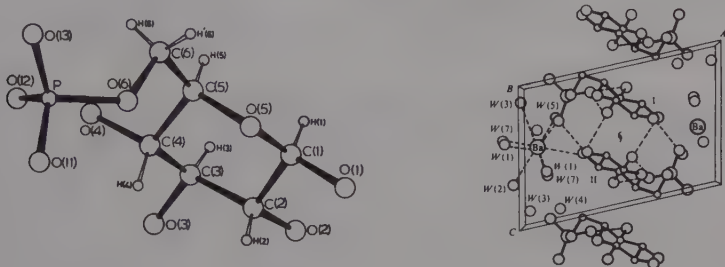


Fig. 1.  $\text{C}_6\text{H}_{11}\text{O}_9\text{P}^{2-} \cdot \text{Ba}^{2+} \cdot 7\text{H}_2\text{O}$ : a perspective view of the anion (left) and the packing arrangement in the unit cell viewed along  $b$  (right).

## 2,4-DIOXA-3-OXO-3-PHOSPHA-3-PHENOXY-trans-BICYCLO[4.4.0]DECANE



P. VAN NUFFEL, A.T.H. LENSTRA and H.J. GEISE, 1982. Bull. Soc. Chim. Belg., 91, 43-48.

Monoclinic,  $P2_1/n$ ,  $a = 20.59$ ,  $b = 6.587$ ,  $c = 20.67 \text{ \AA}$ ,  $\beta = 114.9^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.117$  for 2737 reflexions.

There are two independent molecules with similar conformations (Fig. 1), an all chair form with  $\text{P}=\text{O}$  axial and  $\text{P}-\text{OR}$  equatorial. Some bond lengths are  $\text{P}=\text{O} \ 1.468$  and  $1.489(4)$ ,  $\text{P}-\text{O} \ 1.526-1.609(6) \text{ \AA}$ .

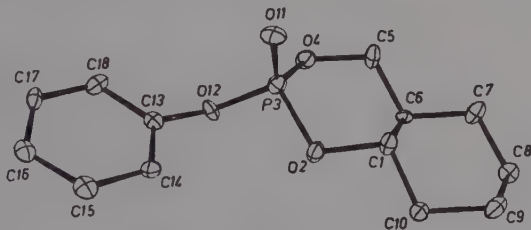
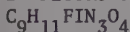


Fig. 1. A view of a  $\text{C}_{13}\text{H}_{17}\text{O}_4\text{P}$  molecule.

## 2'-FLUORO-5-IODOARABINOSYLCYTOSINE



G.I. BIRNBAUM, M. CYGLER, K.A. WATANABE and J.J. FOX, 1982. J. Am. Chem. Soc., 104, 7626-7630.

Monoclinic,  $P2_1$ ,  $a = 4.747$ ,  $b = 14.017$ ,  $c = 18.514 \text{ \AA}$ ,  $\beta = 90.28^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.048$  for 2636 reflexions.





molecules, the fluorine atom is not involved in intermolecular bonding. II differs from normal uridine systems by its unusual sugar puckering C(4')-exo, O(1')-endo (Fig. 1) with  $P = 70.84^\circ$  and  $\tau_m = 38.20^\circ$ . This conformation appears to be stabilised by a close contact between the fluorine atom and the neighbour O(3') proton. Other structural parameters are in the usual range of ribonucleosides: the base is oriented as anti  $\chi = 54.40^\circ$  and the C(4')-C(5') bond orientation is gauche-gauche.

4-(2-DEOXY- $\beta$ -D-ERYTHRO-PENTOFURANOSYL)-6-METHYL-1,2,4-TRIAZIN-3(4H)-ONE 1-OXIDE  
 $C_9H_{13}N_3O_5$

M. BOBEK, M. GLOWKA and R. PARTHASARATHY, 1982. J. Org. Chem., 47, 913-916.

Monoclinic,  $[P2_1]$ ,  $a = 6.832$ ,  $b = 15.550$ ,  $c = 5.109$  Å,  $\beta = 105.29^\circ$ ,  $D_m = 1.54$ ,  $Z = 2$ . Cu radiation,  $R = 0.026$  for 1065 reflexions.

In the molecule (Fig. 1) the bond lengths and angles are in accord with anticipated values. The conformation about the glycosidic bond is anti, and the torsion angle  $\chi_{CN}$  has a very low value  $2.6(8)^\circ$ . The deoxyribose ring is in the twist C(3')-endo/C(2')-exo conformation with O(5') in the  $g^+$  orientation. The  $\phi_{OO}$  and  $\phi_{OC}$  torsional angles are  $-69(1)^\circ$  and  $50(1)^\circ$  respectively. The pseudorotation parameters for the sugar ring are  $P$   $3.9(8)^\circ$  and  $\tau_m$   $36.1(5)^\circ$ , and the displacements of the C(2') and C(3') atoms from the planes through the other four ring atoms are  $-0.519$  and  $0.537$  Å. No stacking between the bases is observed in the crystal. There are two hydrogen bonds, O(5')...O(2) and O(3')...O(4) ( $2.744(4)$  and  $2.843(4)$  Å, respectively).

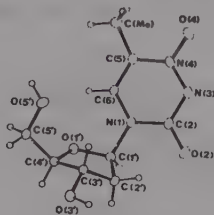


Fig. 1. A view of  $C_9H_{13}N_3O_5$ .

4-AMINO-1- $\beta$ -D-RIBOFURANOSYL-2,6[1H,3H]-PYRIMIDIONE MONOHYDRATE  
 $C_9H_{13}N_3O_6 \cdot H_2O$

J. GORSKI and P. TOLLIN, 1982. Cryst. Struct. Comm., 11, 543-546.

Orthorhombic,  $P2_12_12_1$ ,  $a = 9.020$ ,  $b = 7.02$ ,  $c = 18.69$  Å,  $Z = 4$ . Cu radiation,  $R = 0.051$  for 946 reflexions.

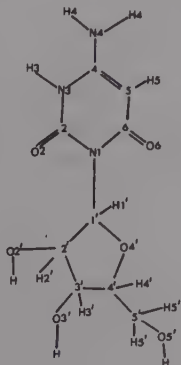
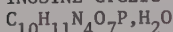


Fig. 1. The  $C_9H_{13}N_3O_6$  molecule.

The analysis shows that N3 is protonated and that O6 exists in the keto form (Fig. 1). The pyrimidine base is essentially planar and the sugar ring is C2' endo. The C5'-O5' bond is in the gauche-gauche orientation relative to C4'-O4' and C4'-C3'. The glycosyl torsion angle  $\chi(\text{CN})$  is in the anti range. All six possible hydrogen donor sites appear to be involved in forming hydrogen bonds or close contacts.

# INOSINE CYCLIC 3',5'-MONOPHOSPHATE MONOHYDRATE



M. SUNDARALINGAM, T.P. HAROMY and P. PRUSINER, 1982. *Acta Cryst.*, **B38**, 1536-1540.

Monoclinic,  $P2_1$ ,  $a = 6.190$ ,  $b = 13.090$ ,  $c = 9.095 \text{ \AA}$ ,  $\beta = 108.43^\circ$ ,  $D_m = 1.656$ ,  $Z = 2$ . Cu radiation,  $R = 0.034$  for 1422 reflexions.

The zwitterion, protonated at N(7) (Fig. 1), has the anti conformation:  $\psi = 18.1(3)^\circ$  and the  $^3T_4$  pucker for the ribofuranose ring:  $P = 27.6(2)^\circ$  and  $\tau_m = 43.4(2)^\circ$ . The phosphate ring has the chair conformation and exhibits the sharpest pucker at the ribose C(3')-C(4') bond. The anti orientation of the base is stabilised by hydrogen bonds to water molecules,  $\text{N}(3) \dots \text{O}(\text{W})$  2.986(3) and  $\text{O}(2') \dots \text{O}(\text{W})$  2.743(4) Å. N(7) is involved in a strong hydrogen bond to a phosphate O of an adjacent nucleotide,  $\text{N}(7) \dots \text{O}(2\text{P})$  2.521(3) Å. The molecular geometry is similar to neutral inosine structures except in the region of N(7) i.e. C(5)-N(7)-C(8) 107.6(3), C(4)-C(5)-N(7) 107.9(3), N(7)-C(8)-N(9) 110.8(3)°. The C(8)-N(9) bond length is 1.349(3) Å. C(1') is 0.153 Å from the least-squares plane of the nine-membered ring while O(6) is displaced 0.016 Å on the opposite side.

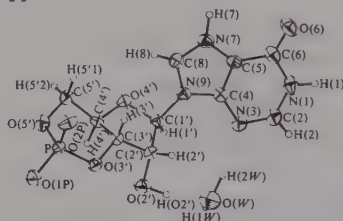
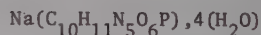
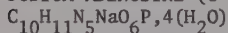


Fig. 1. The  $\text{C}_{10}\text{H}_{11}\text{N}_4\text{O}_7\text{P}, \text{H}_2\text{O}$  structure.

# SODIUM ADENOSINE-(3',5')-CYCLIC-MONOPHOSPHATE TETRAHYDRATE



K.I. VARUGHESE, C.T. LU and G. KARTHA, 1982. *J. Am. Chem. Soc.*, **104**, 3398-3401.

Monoclinic,  $P2_1$ ,  $a = 13.949$ ,  $b = 21.406$ ,  $c = 5.825 \text{ \AA}$ ,  $\beta = 95.47^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.037$  for 3279 reflexions (absolute configuration)

The two independent molecules in the asymmetric unit form a dimer through two  $\text{O}(2')\text{-H} \dots \text{H}(3)$  hydrogen bonds. Conformation about the C(1')-N(9) bond is anti for both molecules, with  $\chi_{\text{CN}}$  values of 31 and 11°. The furanose ring conformations are C(3')endo C(4')exo in one molecule and C(3')endo C(2')exo in the other. The crystal packing is strongly influenced by the cations, the  $\text{Na}^+$  ions being 6-coordinate with Na-O 2.354-2.734 Å. Bond distances ( $\sigma = 0.003$ -0.005 Å) and angles ( $\sigma = 0.25^\circ$ ) for the anion are shown in Fig. 1.

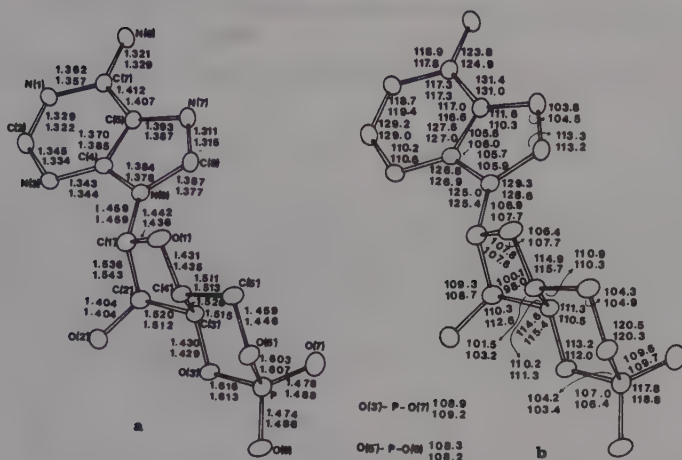


Fig. 1. Bond lengths and angles in the  $(C_{10}H_{11}N_5O_6P)^-$  anion, upper and lower values are for the two independent anions.

8-BROMO-9- $\beta$ -D-XYLOFURANOSYLADENINE HYDRATE  
 $C_{10}H_{12}BrN_5O_4 \cdot 0.67H_2O$

G.I. BIRNBAUM, M. CYGLER, I. EKIEL and D. SHUGAR, 1982. J. Am. Chem. Soc., 104, 3957-3964.

Triclinic,  $P1$ ,  $a = 8.946$ ,  $b = 16.510$ ,  $c = 7.140$  Å,  $\alpha = 90.76^\circ$ ,  $\beta = 89.10^\circ$ ,  $\gamma = 103.72^\circ$ ,  $D_m = 1.760$ ,  $Z = 3$ . Cu radiation,  $R = 0.037$  for 4200 reflexions (absolute configuration).

All three of the crystallographically independent molecules adopt the syn conformation about the glycosidic bond. In two of the molecules the xylose ring has the  $C(4')\text{exo}-C(3')\text{endo}$  pucker with an intramolecular  $O(3')\cdots H\cdots N(3)$  bond ( $O\cdots N$  2.756(6) and 2.770(6) Å) while the third molecule has a  $C(2')\text{endo}$  pucker with an intramolecular  $O(5')\cdots H\cdots N(3)$  bond ( $O\cdots N$  2.856(6) Å). The two different types of molecules are shown in Fig. 1.

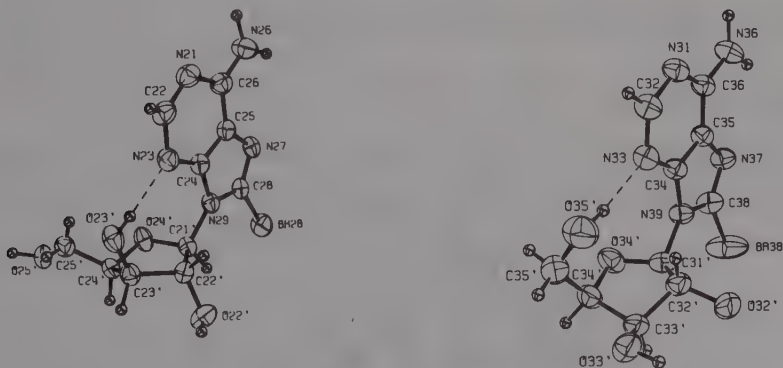


Fig. 1. Views of two of the independent  $C_{10}H_{12}BrN_5O_4$  molecules.

8,2'-ANHYDRO-8-MERCAPTO-9- $\beta$ -D-ARABINOFURANOSYLADENINE 3'-MONOPHOSPHATE  
 $C_{10}H_{12}N_5O_6PS \cdot 2H_2O$

A. MIYAMAE, K. TANAKA, K. HAMADA, S. FUJII and K.-I. TOMITA, 1982. Acta Cryst., B38, 1937-1942.

Monoclinic,  $P2_1$ ,  $a = 11.239$ ,  $b = 11.564$ ,  $c = 13.058$  Å,  $\beta = 110.25^\circ$ ,  $D_m = 1.581$ ,  $Z = 4$ . Cu radiation,  $R = 0.059$  for 2779 reflections.

The conformations of the two zwitterionic molecules in the asymmetric unit are similar except for the orientation of the phosphate groups (Fig. 1) i.e.  $\psi_{CN}$  110.9(8) and 109.7(8) $^\circ$  (both in the high-anti region), both sugar puckerings are C(4')-endo with C(4') displaced 0.42(1) and 0.41(2) Å from the C(3')-C(2')-C(1')-O(1') plane, the orientation of the C(5')-O(5') bond is gauche-trans but the  $\phi'$  torsion angles are 191.9(6) and 255.4(7) $^\circ$ . Phosphate and water molecules are linked by a network of hydrogen bonds with all available H atoms participating; the bonds N(1)-H...O(6) 2.610 and 2.666(10) and O(7)-H...O(W) 2.591 and 2.572(14) are strong.

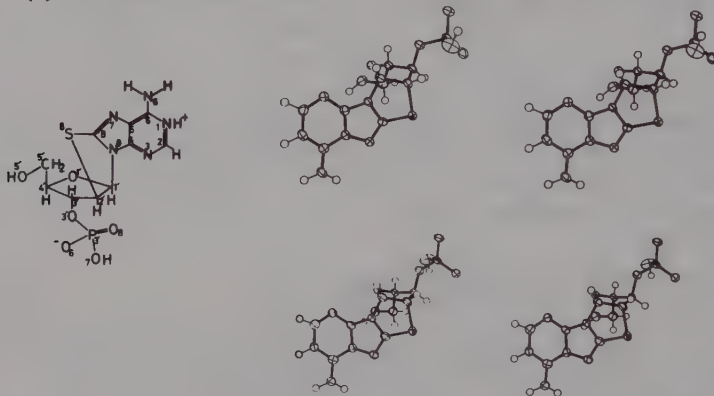


Fig. 1. The chemical structure and stereoviews of the two independent  $C_{10}H_{12}N_5O_6PS$  molecules.

1-(3'-C-METHYL- $\beta$ -D-RIBOFURANOSYL)CYTOSINE  
 $C_{10}H_{15}N_3O_5$

G.V. GURSKAYA, G.M. JAVADOVA and S.N. MIKHAILOV, 1982. Cryst. Struct. Comm., 11, 1253-1258.

Orthorhombic,  $P2_12_12_1$ ,  $a = 10.874$ ,  $b = 11.090$ ,  $c = 9.799$  Å,  $Z = 4$ . Cu radiation,  $R = 0.062$  for 1207 reflexions.

The cytosine moiety of the molecule (Fig. 1) is nearly planar. The conformation of the ribose is C(2')-endo-C(3')-exo,  $^2T_3$ . The relative orientation of the base and ribose planes corresponds to the anti-conformation.

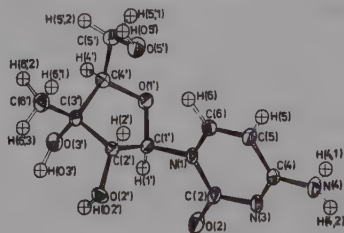


Fig. 1. A view of  $C_{10}H_{15}N_3O_5$ .

1-(6-DEOXY- $\beta$ -D-ALLOFURANOSYL)CYTOSINE

G.V. GURSKAYA, A.S. ZHDANOV, S.N. MIKHAILOV and E.N. TSAPKINA, 1982. Cryst. Struct. Comm., 11, 1245-1252.

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.979$ ,  $b = 11.792$ ,  $c = 12.659$  Å,  $Z = 4$ . Cu radiation,  $R = 0.039$  for 1357 reflexions.

The atoms of the cytosine moiety (Fig. 1) are nearly coplanar, and the ribose group has the C(2')-endo-C(3')-exo conformation  ${}^2T_3$ . The relative orientation of the base and ribose planes is anti. The torsion angle O(1')-C(1')-N(1)-C(6) is  $54.5^\circ$ , and the conformation about the C(4')-C(5') is gauche-gauche. All possible hydrogen bonds are utilised in the crystal structure.

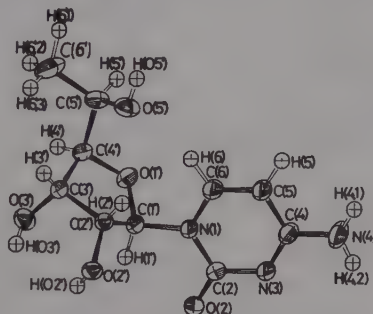
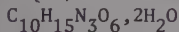


Fig. 1. A view of  $\text{C}_{10}\text{H}_{15}\text{N}_3\text{O}_5$ .

1-(2- $\beta$ -D-PSICOFURANOSYL)CYTOSINE DIHYDRATE

G.V. GURSKAYA, G.M. JAVADOVA, S.G. ZAVGORODNY, T.L. TSILEVICH and B.P. GOTTIKH, 1982. Cryst. Struct. Comm., 11, 1259-1264.

Orthorhombic,  $P2_12_12$ ,  $a = 7.719$ ,  $b = 24.691$ ,  $c = 7.010$  Å,  $Z = 4$ . Cu radiation,  $R = 0.040$  for 1510 reflexions.

The cytosine moiety of the molecule (Fig. 1) is nearly planar and the ribose conformation is C(3')-endo-C(2')-exo  ${}^3T_2$ . Base and ribose orientation corresponds to anti. The presence of a hydroxymethyl substituent at (C1') causes no basic change in the structure of the cytidine residue.

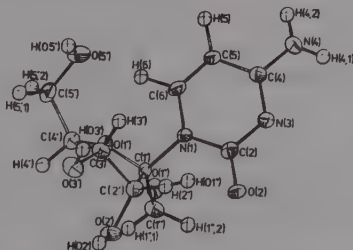


Fig. 1. A view of  $\text{C}_{10}\text{H}_{15}\text{N}_3\text{O}_6$ .





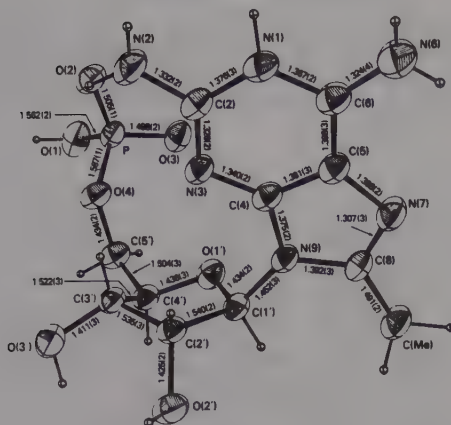
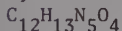


Fig. 1. Bond lengths and absolute configuration of  $C_{11}H_{14}N_7O_7P$ .

1,N<sup>6</sup>-ETHENOADENOSINE

M. JASKOLSK, 1982. *Acta Cryst.*, B38, 3171-3174.

Orthorhombic,  $P2_12_12_1$ ,  $a = 6.4679$ ,  $b = 10.4029$ ,  $c = 18.277$  Å,  $Z = 4$ . Cu radiation,  $R = 0.037$  for 977 reflexions.

The adenine moiety is not planar but has a U shape (Fig. 1). The ribose ring is 2'-endo puckered with a  $g^+$  side chain. The glycosidic side chain has an unusual value ( $26.2(4)^\circ$ , anti), which is rare in  $\beta$ -ribofuranosides. There is almost no base overlap in the structure. Molecules are linked by a three-dimensional hydrogen-bond network.

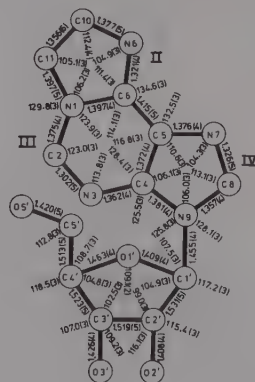
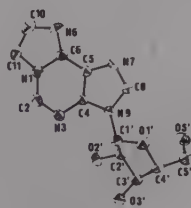
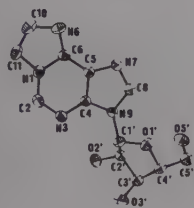
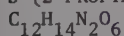


Fig. 1. A stereoview of  $C_{12}H_3N_5O_4$  and details of dimensions.

## 5-(2-PROPYNYLOXY)-2'-DEOXYURIDINE



G.S.D. KING and L. SENGLER-ROBERTS, 1982. J. Chem. Research, S, 25; M, 0722-0744.

Monoclinic,  $P2_1$ ,  $a = 10.331$ ,  $b = 13.706$ ,  $c = 4.842$  Å,  $\beta = 103.54^\circ$ ,  $D_m = 1.41$ ,  $Z = 2$ .  
Mo radiation,  $R = 0.027$  for 1060 reflexions.

The propynyloxy side-chain lies in a plane with C(5) which makes a dihedral angle of  $7.7^\circ$  with the plane of the pyrimidine ring. The deoxypentose has the C(2')-endo conformation and the molecule as a whole has the C(2')-endo-gauche-anti conformation. Molecules in the crystal are linked by H bonds from O(5')-H to O(4) and O(3')-H to O(2) (both 2.77 Å).

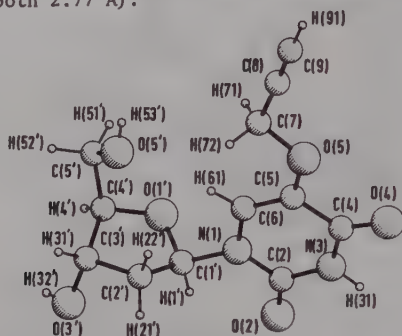
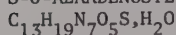


Fig. 1.  $C_{12}H_{14}N_2O_6$ : the molecular structure.

# S-8-AZAADENOSYL-L-HOMOCYSTEINE



V.B. PETT, H.-S. SHIEH and H.M. BERMAN, 1982. *Acta Cryst.*, **B38**, 2611-2615.

Orthorhombic,  $P2_12_12_1$ ,  $a = 5.154$ ,  $b = 17.672$ ,  $c = 38.019$  Å,  $Z = 8$ . Cu radiation,  $R = 0.091$  for 1706 reflexions.

The conformations of the two independent molecules are different (Fig. 1). Molecule A has C(2')-endo sugar pucker, and a 'high anti' glycosyl ( $\chi$ ) torsion angle of  $114^\circ$ , while molecule B has a C(2')-exo-C(3')-endo sugar pucker and  $\chi$  angle of  $42^\circ$ . The crystal structure consists of two types of hydrogen-bonded columns; one contains the bases and ribose rings and the other contains the amino acids, sugars and water molecules. It is concluded that the two molecules are dipolar ions, with negatively charged carboxyl and positively charged amino groups.

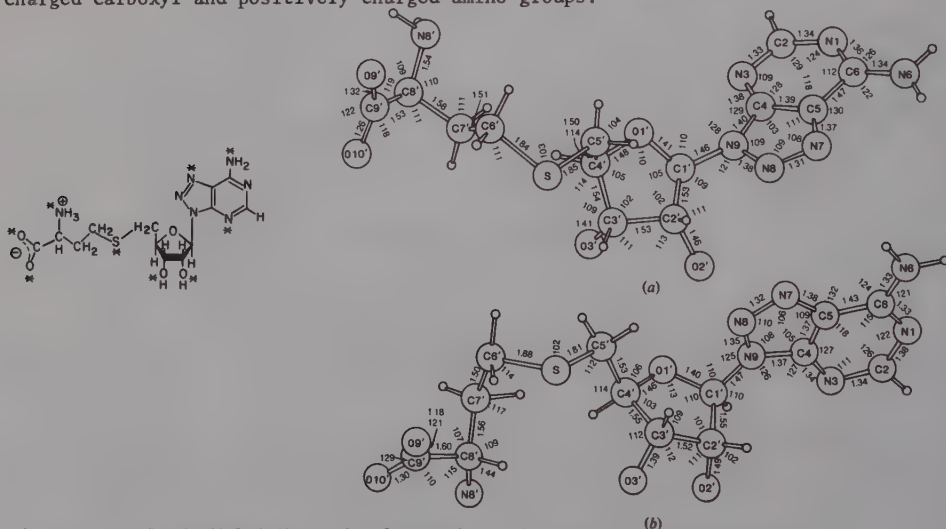


Fig. 1.  $C_{13}H_{19}N_7O_5S \cdot H_2O$ : bond lengths and angles in (a) molecule A, and (b) molecule B.

5-ACETYL-1-(3,5-O-ISOPROPYLIDENE- $\beta$ -D-XYLOFURANOSYL)URACIL  
 $C_{14}H_{18}N_2O_7$

D.W. JONES and J.M. SOWDEN, 1982. *Acta Cryst.*, B38, 1962-1965.

Orthorhombic,  $P2_12_12_1$ ,  $a = 6.135$ ,  $b = 9.227$ ,  $c = 26.11$  Å,  $D_m = 1.44$ ,  $Z = 4$ . Cu radiation,  $R = 0.048$  for 1684 reflexions.

The molecule (Fig. 1) has a slightly non-planar (maximum displacement 0.03(2) Å - imido N(2)) uracil ring  $\beta$ -linked to a C(3')-endo, C(4')-exo ( $^3T_4$  puckered) xylofuranose ring. The angle C(3)-C(2)-O(2) is 127.9(3) while N(2)-C(2)-O(2) is 118.2(3) and C(1)-N(2)-C(2) is 126.8(3)°. External angles at the glycosidic linkage are C(1)-N(1)-C(7) 115.4(2) and C(4)-N(1)-C(7) 123.1(2)° and the dihedral angle between the pyrimidine and sugar ring planes is 73°. The sugar/base orientation angle between O(4)-C(7) and N(1)-C(4) measured along C(7)-N(1) is 16.9(4)°. Molecules are linked by an O(5)...O(3) hydrogen bond of length 2.715(4) Å.

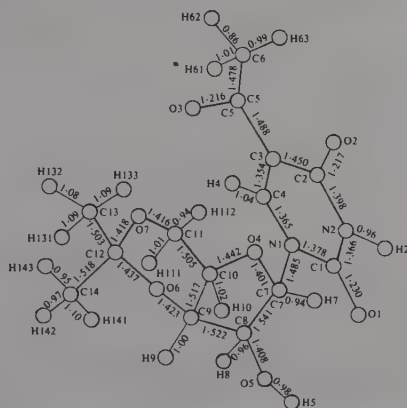
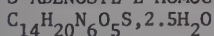


Fig. 1. Bond lengths (e.s.d.s. for non-hydrogen atom bonds 0.004 Å) in  $C_{14}H_{18}N_2O_7$ .

S-ADENOSYL-L-HOMOCYSTEINE HYDRATE



H.-S. SHIEH and H.M. BERMAN, 1982. *Acta Cryst.*, B38, 1513-1517.

Monoclinic,  $C2$ ,  $a = 45.658$ ,  $b = 5.6790$ ,  $c = 15.592$  Å,  $\beta = 99.752^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.074$  for 2329 reflexions.

The two independent molecules (A and B) have different conformations with respect to both amino acid and nucleoside portions (Fig. 1). The glycosidic angle is anti in both but is much larger in A. The sugar puckering is C(2')-endo in A and C(3')-endo in B. The asymmetric carbons of the amino acid portions are in the S configuration in A and B. Both molecules are zwitterions with N(8') protonated. There is an extensive hydrogen-bond network involving all available hydrogen bond donors and acceptors and which includes asymmetric base pairing such that N(6) and N(7) of A bond to N(1) and N(6) of B. Five water molecules per asymmetric unit are found in three positions of full occupancy and four of half occupancy.

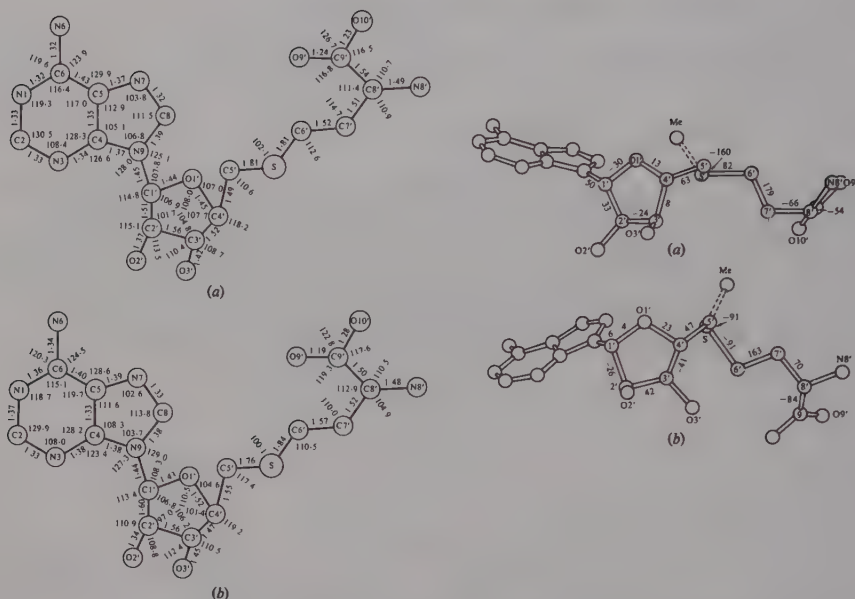


Fig. 1. Bond lengths and angles in  $C_{14}H_{20}N_6O_5S$  and the conformation of the two independent molecules.

8,2'-ANHYDRO-8-MERCAPTO-9- $\beta$ -D-ARABINOFURANSYLHYPOXANTHYLYL-(3'-5')-8,2'-ANHYDRO-8-MERCAPTO-9- $\beta$ -D-ARABINOFURANSYLADENINE HEXAHYDRATE  
 $C_{20}H_{20}N_9O_9PS_2 \cdot 6H_2O$

K. HAMADA, Y. MATSUO, A. MIYAMAE, S. FUJII and K.-I. TOMITA, 1982. *Acta Cryst.*, **B38**, 2528-2531.

Triclinic,  $P1$ ,  $a = 14.324$ ,  $b = 11.130$ ,  $c = 5.794$  Å,  $\alpha = 97.40^\circ$ ,  $\beta = 87.42^\circ$ ,  $\gamma = 120.05^\circ$ ,  $D_m = 1.630$ ,  $Z = 1$ . Cu radiation,  $R = 0.066$  for 2697 reflexions.

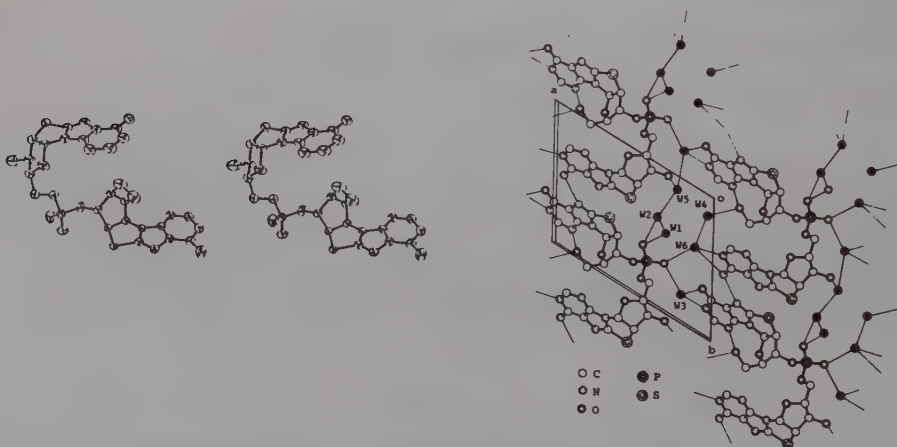


Fig. 1.  $C_{20}H_{20}N_9O_9PS_2 \cdot 6H_2O$ : stereoview of the molecular conformation and a view of the molecular packing.

The molecular conformation (Fig. 1) is a folded form which is stabilised by hydrophobic interactions between sugar and base moieties and by intermolecular base stacking involving O-H...O, O-H...N, N-H...O and N-H...N hydrogen bonds. Bond lengths and angles are generally in agreement with those in related compounds.

8,2'-S-CYCLO-2'-THIOADENYL(3'-5')-8,2'-S-CYCLO-2'-THIOADENOSINE HYDROCHLORIDE  
 $C_{20}H_{22}ClN_{10}O_8PS_2 \cdot 2.5(H_2O)$

S. FUJII, K. HAMADA, R. MIURA, S. UESUGI, M. IKEHARA and K.-I. TOMITA, 1982. Acta Cryst., B38, 564-570.

Triclinic, P1,  $a = 11.161$ ,  $b = 11.824$ ,  $c = 12.136$  Å,  $\alpha = 89.20^\circ$ ,  $\beta = 97.92^\circ$ ,  $\gamma = 116.75^\circ$ ,  $D_m = 1.655$ ,  $Z = 2$ . Mo radiation,  $R = 0.078$  for 4976 reflexions.

The two independent molecules are in almost identical sharp "bend" conformations (Fig. 1) i.e. rotation around P-O is ( $g^-, t$ ). The dihedral angles between bases in the same molecule are  $63.6$  and  $59.4(4)^\circ$ . Sugar puckerings are C(4')-endo (5'-end side) and C(3')-exo (3'-end side) in both molecules. There is extensive hydrogen bonding linking ions and water molecules in which all hydrogen donor atoms participate. There is no intramolecular base stacking or base pairing but there is strong intermolecular stacking between bases at the 5'-end sides with average separation of  $3.4$  Å. The base moiety at the 3'-end side has no stacking.

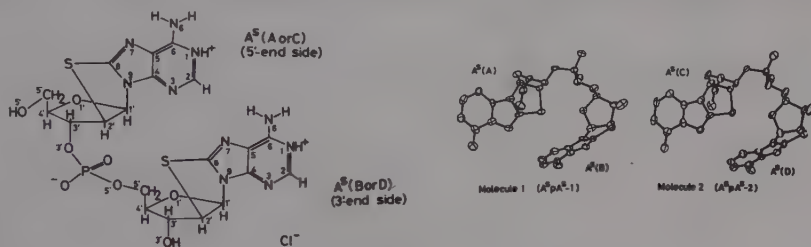


Fig. 1. The  $C_{20}H_{22}N_{10}O_8PS_2^+$  ions.

#### PHOSPHONOMETHYLGLYCINE

$C_3H_8NO_5P$  (I)

$(HO)_2PCH_2NH_2CH_2COOH$

#### IMINODIACETATOMETHYLPHOSPHONIC ACID

$C_5H_{10}NO_7P$  (II)

$(HO)_2PCH_2NH(CH_2COOH)_2$

L.M. SHKOL'NIKOVA, M.A. PORAI-KOSHITS, N.M. DYATLOVA, G.F. YAROSHENKO, M.V. RUDOMINO and E.K. KOLOVA, 1982. Zh. Strukt. Khim., 23-5, 98-107 [J. Struct. Chem., 23, 737-746].

I. Monoclinic,  $P2_1/c$ ,  $a = 8.681$ ,  $b = 7.981$ ,  $c = 9.893$  Å,  $\beta = 105.77^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.035$  for 1712 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 5.590$ ,  $b = 7.422$ ,  $c = 10.648$  Å,  $\alpha = 93.12^\circ$ ,  $\beta = 95.03^\circ$ ,  $\gamma = 90.40^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.050$  for 1113 reflexions.

The two molecules (Fig. 1) have similar geometric parameters. The nitrogen atoms are protonated by a proton of the phosphonic acid group, rather than by a proton of a carboxylic acid function. Bond lengths are normal for both molecules ( $\sigma = 0.001-0.002$  Å and  $0.003-0.007$  Å for the nonhydrogen atoms of I and II respectively).



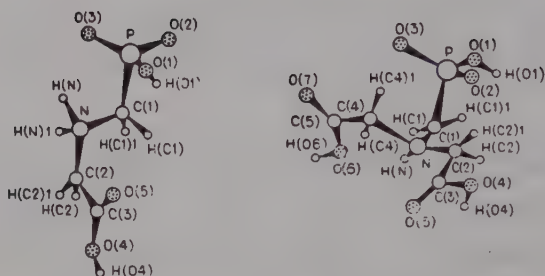


Fig. 1. The molecules  $(\text{HO})\text{O}_2\text{PCH}_2\text{NH}_2\text{CH}_2\text{COOH}$  (left) and  $(\text{HO})\text{O}_2\text{PCH}_2\text{NH}(\text{CH}_2\text{COOH})_2$  (right).

### L-HOMOSERINE



K.K. CHACKO, S. SWAMINATHAN and K.R. VEENA, 1982. Cryst. Struct. Comm., **11**, 2057-2062.

Orthorhombic,  $\text{P2}_1\text{2}_1\text{2}_1$ ,  $a = 9.189$ ,  $b = 11.565$ ,  $c = 5.435 \text{ \AA}$ ,  $D_m = 1.36$ ,  $Z = 4$ . Cu radiation,  $R = 0.071$  for 581 reflexions.

The molecule (Fig. 1) is a zwitterion with  $\psi^1 -33.3$ ,  $\chi^1 -174.0$  and  $\chi^2 70.5^\circ$ . Molecules are linked by N-H...O and O-H...O hydrogen bonds (2.73 to 2.80  $\text{\AA}$ ).

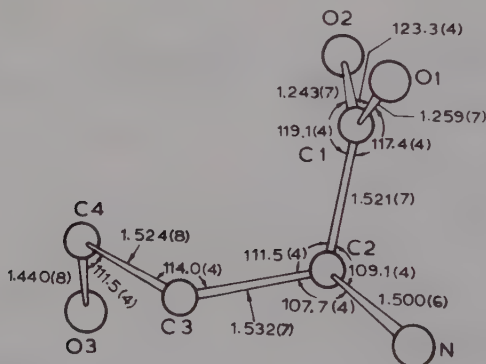
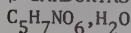


Fig. 1. L-homoserine and dimensions.

### $\beta$ -CARBOXYASPARTIC ACID HYDRATE



B. RICHEY, M.R. CHRISTY, R.C. HALTIWANGER, T.H. KOCH and S.J. GILL, 1982. Biochemistry, **21**, 4819-4823.

Monoclinic,  $\text{Cc}$ ,  $a = 13.112$ ,  $b = 8.207$ ,  $c = 7.292 \text{ \AA}$ ,  $\beta = 108.03^\circ$ ,  $D_m = 1.73$ ,  $Z = 4$ . Mo radiation,  $R = 0.029$  for 765 reflexions.

The X-ray data indicate that in the solid, the negative charge of the zwitterion is distributed approximately equally between one of the  $\beta$ -carboxyls and the  $\alpha$ -carboxyl group. Molecules of the acid are linked by very short (2.444  $\text{\AA}$ ) hydrogen bonds between one of the  $\beta$ -carboxyls and the  $\alpha$ -carboxyl of an adjacent molecule (Fig. 1).

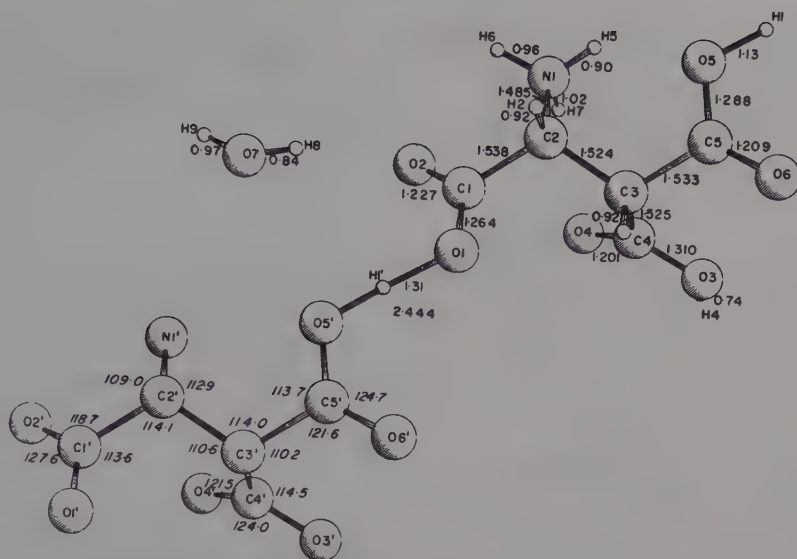


Fig. 1. A drawing of  $\beta$ -carboxyaspartic acid hydrate with dimensions.

#### N-CARBAMOYL-DL-ASPARTIC ACID



G.S. JAGANNATHA RAO, H.M. KRISHNA MURTHY, N. APPAJI RAO and M. VIJAYAN, 1982. Acta Cryst., B38, 1672-1674.

Triclinic,  $P\bar{1}$ ,  $a = 6.438$ ,  $b = 7.486$ ,  $c = 8.048$  Å,  $\alpha = 72.2^\circ$ ,  $\beta = 80.8^\circ$ ,  $\gamma = 76.4^\circ$ ,  $D_m = 1.65$ ,  $Z = 2$ . Cu radiation,  $R = 0.095$  for 1205 reflexions.

In the molecule (Fig. 1), the unionised  $\alpha$ -carboxyl group C(2), and N(1) lie approximately in a plane. The planes of the  $\alpha$ -carboxyl and ureido groups are inclined at  $14.8^\circ$ . The side chain assumes the sterically least favourable conformation with its unionised carboxyl group staggered between the  $\alpha$ -carboxyl group and N(1). The structure is stabilised by hydrogen bonds i.e. O(2)...O(3) 2.59(1), O(5)...O(3) 2.69(1), N(1)...O(4) 2.98(1), N(2)...O(4) 3.07(1), N(2)...O(1) 2.98 Å. The N(2)...O(1) and O(2)...O(3) interactions are nearly parallel.

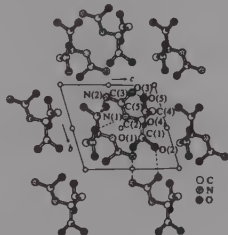
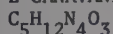


Fig. 1. The crystal structure of N-carbamoyl-DL-aspartic acid.

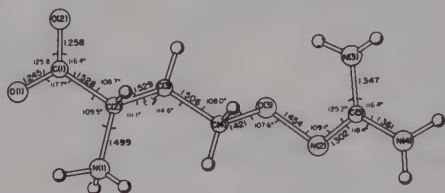
#### L-CANAVANINE



A. BOYAR and R.E. MARSH, 1982. J. Am. Chem. Soc., 104, 1995-1998.

Monoclinic,  $P2_1$ ,  $a = 5.505$ ,  $b = 8.419$ ,  $c = 8.432$  Å,  $\beta = 93.06^\circ$ ,  $D_m = 1.494$ ,  $Z = 2$ . Cu radiation,  $R = 0.040$  for 827 reflexions.

The zwitterionic molecule (Fig. 1) is protonated on the  $\alpha$ -amino nitrogen; the neutral guanidine grouping is in the amino form, which seems to be preferred by all guanidine groups with electron-withdrawing substituents.



There are two independent molecules showing flat backbone conformation (Fig. 1), which include weak intramolecular bifurcated hydrogen bonds. Strong intermolecular hydrogen bonds determine the crystal packing, in which antiparallel chains of single molecules run along [010].

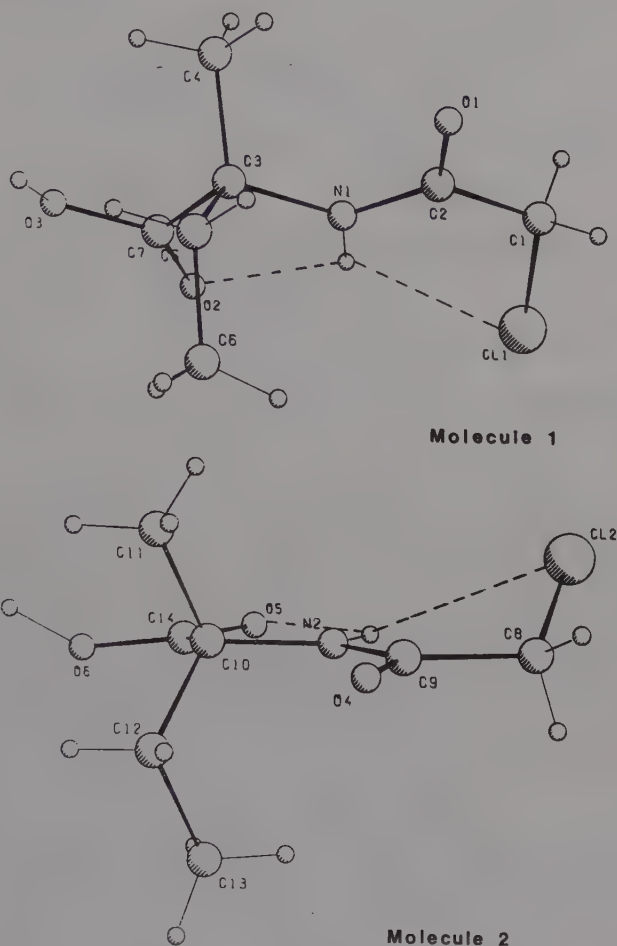
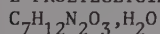


Fig. 1. The two independent  $C_7H_{12}ClNO_3$  molecules.

#### L-PROLYLGLYCINE MONOHYDRATE



P. NARASIMHAN and K.K. CHACKO, 1982. Cryst. Struct. Comm., 11, 2051-2056.

Monoclinic, P2,  $a = 6.553$ ,  $b = 5.417$ ,  $c = 13.262$  Å,  $\beta = 101.26^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.093$  for 1080 reflexions.

The molecule (Fig. 1) is a zwitterion with N(1) protonated; atom C3 is



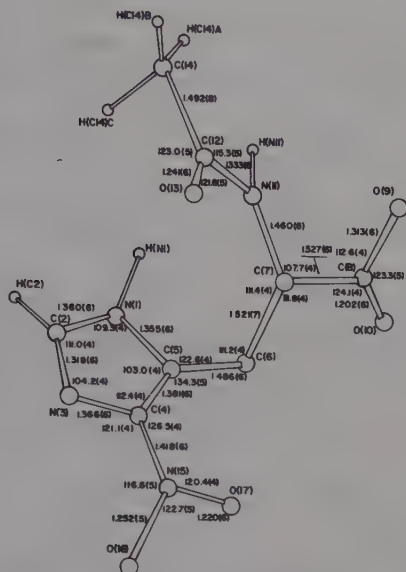
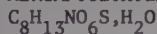


Fig. 1. Bond lengths (Å) and angles (°) in the N<sup>α</sup>-acetyl-5-nitro-L-histidine molecule.

MERCAPTOETHYLAMINETRIACETIC ACID MONOHYDRATE



L.M. SHKOL'NIKOVA, V.Pa. TEMKINA, N.M. DYATLOVA and J. PODLAHA, 1982. Zh. Strukt. Khim., 23-1, 135-139 [J. Struct. Chem., 23, 111-114].

Triclinic,  $P\bar{1}$ ,  $a = 5.669$ ,  $b = 10.366$ ,  $c = 10.842$  Å,  $\alpha = 102.14$ ,  $\beta = 96.26$ ,  $\gamma = 102.60^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.042$  for 2764 reflexions.

The molecule (Fig. 1) exists as a zwitterion, the quaternary NH proton forming a pair of intramolecular N-H...O hydrogen bonds. The S-C-C-N torsion angle is  $\sim 54^\circ$ . The molecules form chains along the y axis via O-H...O hydrogen bonding involving all of the carbonyl groups and the water molecule (see Fig. 1). Bond lengths ( $\sigma \approx 0.003$ - $0.005$  Å) are normal.

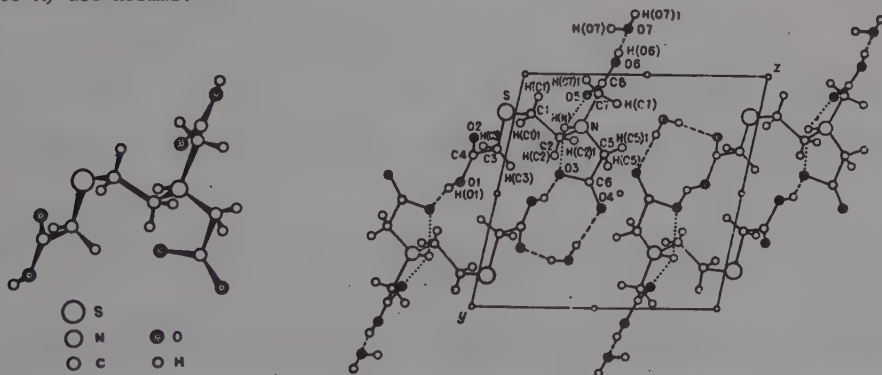
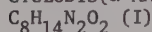
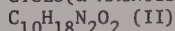


Fig. 1. The structure of  $C_8H_{13}NO_6S \cdot H_2O$ .



CYCLOBIS( $\alpha$ -AMINOISOBUTYRYL)CYCLO( $\alpha$ -AMINOISOBUTYRYL-ISOLEUCYL)

K. SUGUNA, S. RAMAKUMAR, N. SHAMALA, B.V.V. PRASAD and P. BALARAM, 1982. Biopolymers, 21, 1847-1855.

I. Triclinic,  $P\bar{1}$ ,  $a = 5.649$ ,  $b = 5.865$ ,  $c = 8.363 \text{ \AA}$ ,  $\alpha = 69.89$ ,  $\beta = 113.04$ ,  $\gamma = 116.0^\circ$ ,  $Z = 1$ . Cu radiation,  $R = 0.085$  for 726 reflexions.

II. Orthorhombic,  $P2_12_12_1$ ,  $a = 6.177$ ,  $b = 10.791$ ,  $c = 16.676 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.086$  for 1022 reflexions.

The diketopiperazine ring in both structures (Fig. 1) shows small deviations from planarity. Molecule I lies about an inversion centre and has a very flat chair conformation with C $\alpha$  atoms 0.07  $\text{\AA}$  from the plane of the other four ring atoms. Molecule II has a slight boat conformation, with Aib C $\alpha$  and Ile C $\alpha$  atoms 0.11 and 0.05  $\text{\AA}$  respectively from the plane of the remaining four atoms.

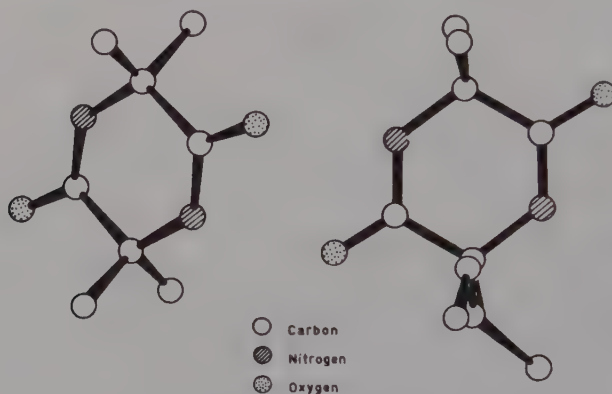


Fig. 1. Views of  $C_8H_{14}N_2O_2$  (left) and  $C_{10}H_{18}N_2O_2$  (right).

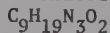
## ACETYL-L-VALINE DIMETHYLAMIDE



A. AUBRY, M.T. CUNG and M. MARRAUD, 1982. Cryst. Struct. Comm., 11, 129-134.

Orthorhombic,  $P2_12_12_1$ ,  $a = 4.854$ ,  $b = 9.775$ ,  $c = 23.006 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.059$  for 932 reflexions.

The molecules have normal dimensions and are hydrogen bonded to form chains along  $a$ , with  $N \cdots O$  2.906(6)  $\text{\AA}$ .

 $N^\alpha$ -ACETYL-AZA- $\alpha'$ -HOMO-L-VALINE DIMETHYLAMIDE

A.F. MISHNEV, J. BLEIDELIS, J.E. ANCANS and G.I. CIPENS, 1982. Zh. Strukt. Khim., 23-2, 101-106 [J. Struct. Chem., 23, 252-256].

Orthorhombic,  $P2_12_12_1$ ,  $a = 9.005$ ,  $b = 13.925$ ,  $c = 19.295 \text{ \AA}$ ,  $Z = 8$ . Cu radiation,  $R = 0.083$  for 1400 reflexions.

The two crystallographically independent molecules (Fig. 1) have different conformations; the angles  $\phi$ ,  $\psi$ ,  $\omega_1$ , and  $\omega_2$  being  $-121$ ,  $90$ ,  $179$ , and  $179^\circ$  for molecule A and  $-105$ ,  $124$ ,  $173$ , and  $179^\circ$  for molecule B. Molecules are joined in layers by N-H...O hydrogen bonding. Bond lengths ( $\sigma = 0.01$ - $0.02$  Å) are normal.

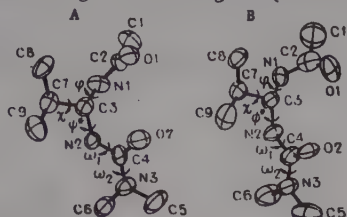


Fig. 1. The two crystallographically independent  $C_9H_{19}N_3O_2$  molecules.

N-(N-ACETYL-L-ASPARTYL)-4-AMINOBUTYRIC ACID  
 $C_{10}H_{16}N_2O_6$

L.K. HANSEN, E.A. HAGEN, T. LOENNECHEN and A.J. AASEN, 1982. Acta Chem. Scand., B36, 327-335.

Monoclinic,  $P2_1$ ,  $a = 4.720$ ,  $b = 15.242$ ,  $c = 8.383$  Å,  $\beta = 93.19^\circ$ ,  $D_m = 1.45$ ,  $Z = 2$ .  
 Mo radiation,  $R = 0.066$  for 1512 reflexions (at  $-150^\circ\text{C}$ ).

The molecule has an open structure with no intramolecular contacts (Fig. 1). It can best be described in terms of four planes, P1: O(101), O(102), C(9), C(8) and C(7); P2: C(7), N(2), C(6), O(6) and C(3); P3: C(6), C(3), C(4), C(5), O(51) and O(52); and P4: C(3), N(1), C(2), O(2) and C(1). P1 and P4 have an angle of  $18.5^\circ$  between them, P2 and P3  $84.2^\circ$ , P2 and P4  $84.4^\circ$  and P3 and P4  $74.3^\circ$ . Relevant bond lengths are: N(1)-C(3) 1.464, C(3)-C(4) 1.521, C(4)-C(5) 1.529, C(5)-O(51) 1.172, C(5)-O(52) 1.329, C(3)-C(6) 1.524, C(6)-O(6) 1.247, N(2)-C(7) 1.456, C(7)-C(8) 1.530, C(8)-C(9) 1.523, C(9)-C(10) 1.499, C(10)-O(101) 1.219 and C(10)-O(102) 1.332 Å.

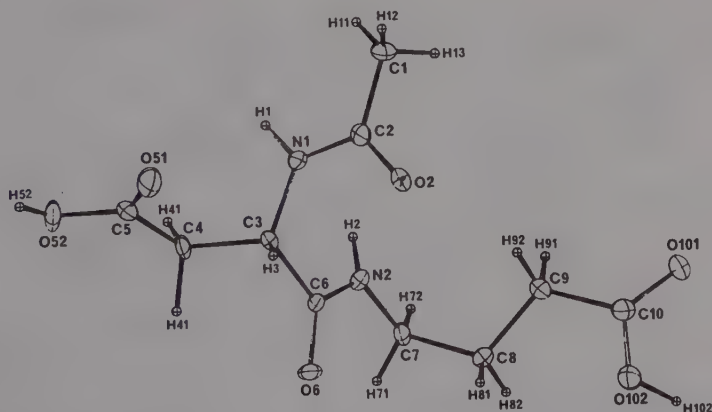


Fig. 1. Molecular structure of  $C_{10}H_{16}N_2O_6$ .

$\alpha$ -L-GLUTAMYL-L-GLUTAMIC ACID  
 $C_{10}H_{16}N_2O_7$

D.S. EGGLESTON and D.J. HODGSON, 1982. Acta Cryst., B38, 1216-1220.

Monoclinic,  $P2_1$ ,  $a = 5.343$ ,  $b = 7.141$ ,  $c = 15.944$  Å,  $\beta = 90.11^\circ$ ,  $D_m = 1.51$ ,  $Z = 2$ . Mo radiation,  $R = 0.050$  for 1632 reflexions.

The dipeptide (Fig. 1) occurs as a zwitterion in the crystal, with the amino terminus protonated and the main-chain carboxyl group deprotonated. The two glutamyl side chains are on opposite sides of the backbone, and the structure is extended. The peptide linkage is significantly non-planar, the  $\omega$  torsional angle being  $167.6^\circ$ . There is extensive intermolecular hydrogen bonding in the crystals, but no intra-molecular hydrogen bonding.

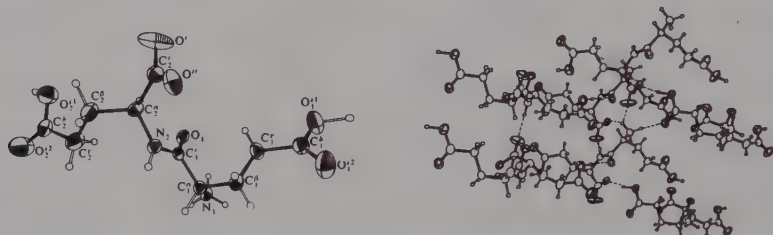


Fig. 1.  $C_{10}H_{16}N_2O_7$ : a perspective view of the molecule (left) and packing and hydrogen bonding in the crystal (right).

#### L-PROLYL-L-VALINE MONOHYDRATE

$C_{10}H_{18}N_2O_3 \cdot H_2O$

P. NARASIMHAN, K.K. CHACKO and S. SWAMINATHAN, 1982. Cryst. Struct. Comm., **11**, 695-700.

Orthorhombic,  $P2_12_12_1$ ,  $a = 6.549$ ,  $b = 35.485$ ,  $c = 5.436$  Å,  $Z = 4$ . Cu radiation,  $R = 0.088$  for 1014 reflexions.

The molecule (Fig. 1) exists as a zwitterion with N(1) protonated and the valine carboxyl ionised. There is disorder of C3 of the pyrrolidine ring. Molecules are linked by N-H...O and O-H...O hydrogen bonds.

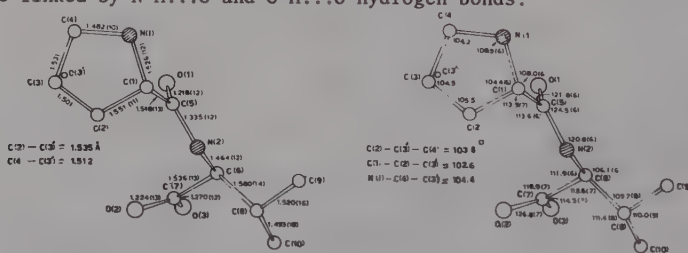


Fig. 1. Bond lengths and angles in L-prolyl-L-valine monohydrate.

#### L-ARGININE L-ASPARTATE

$C_{10}H_{21}N_5O_6$

$C_6H_{15}N_4O_2^+ \cdot C_4H_6NO_4^-$

D.M. SALUNKE and M. VIJAYAN, 1982. Acta Cryst., **B38**, 1328-1330.

Monoclinic,  $P2_1$ ,  $a = 5.511$ ,  $b = 8.438$ ,  $c = 15.265$  Å,  $\beta = 97.9^\circ$ ,  $D_m = 1.467$ ,  $Z = 2$ . Cu radiation,  $R = 0.044$  for 1226 reflexions.

The crystal structure is made up of positively charged arginine and negatively charged aspartate ions and is stabilised by ionic interactions and hydrogen bonds (Fig. 1). Bond lengths and angles are comparable to those in similar structures.

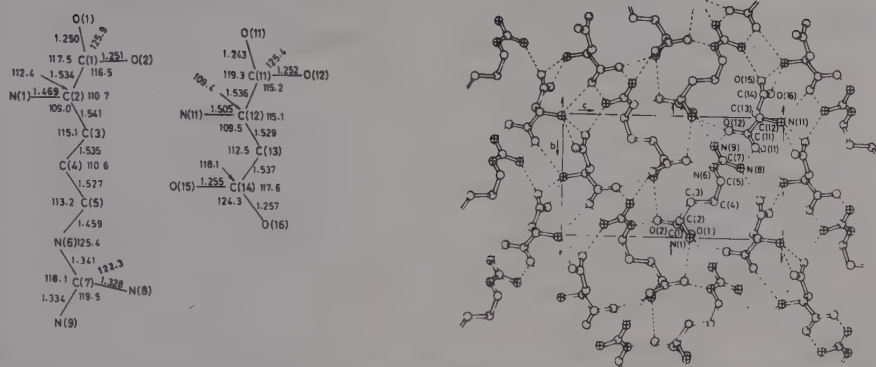


Fig. 1.  $C_{10}H_{21}N_5O_6$ : bond lengths and angles (left) and the crystal structure as viewed along the a axis, with broken lines indicating hydrogen bonds (right).

# $N^\alpha$ -DITHIASUCCINOYL L-PHENYLALANINE



G. BARANY, 1982. Cryst. Struct. Comm., **11**, 913-928.

Monoclinic,  $C_2$ ,  $a = 25.007$ ,  $b = 9.372$ ,  $c = 11.115$  Å,  $\beta = 102.82^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.046$  for 4032 reflexions.

The asymmetric unit (Fig. 1) consists of two independent molecules linked through pairs of hydrogen bonds. The molecules differ in conformation but have no differences in bond lengths. A comparison is made with other cyclic disulphides.

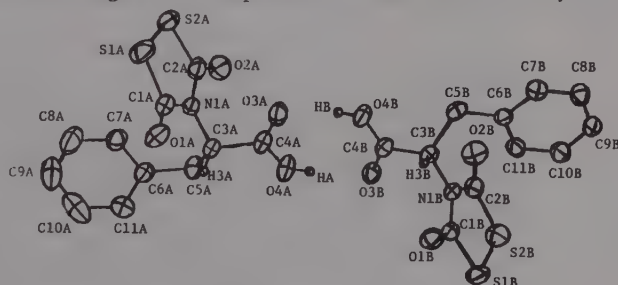
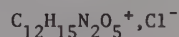
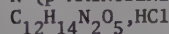


Fig. 1. The asymmetric unit of  $C_{11}H_9NO_4S_2$ .

# $N$ -(p-AMINOBENZOYL)-L-GLUTAMIC ACID HYDROCHLORIDE



C. CHATTERJEE, J.K. DATTA GUPTA and N.N. SAHA, 1982. Acta Cryst., **B38**, 2086-2089.

Monoclinic,  $P2_1$ ,  $a = 11.819$ ,  $b = 4.924$ ,  $c = 12.085$  Å,  $\beta = 102.4^\circ$ ,  $D_m = 1.47$ ,  $Z = 2$ . Cu radiation,  $R = 0.065$  for 1022 reflexions.

The p-aminobenzoic acid part of the molecule is linked to the glutamic acid part through a peptide-like linkage with  $C(7)-N(2)$  1.340(9) Å (Fig. 1). The glutamic acid side chain is buckled with  $C^\alpha$  gauche to  $C^\beta$  ( $\chi^2 = 74.2^\circ$ ) resulting in a close contact of 2.923(9) Å between  $C(12)$  and the amide  $N(2)$ . The  $\alpha$ -carboxyl C atom is trans to  $CY$  the torsion angle  $C-C^\alpha-C^\beta-CY$  being  $-176.5^\circ$ . The  $\alpha$ -N atom is 0.526 Å from the  $\alpha$ -carboxyl group plane and  $\psi^1$  is  $22.5^\circ$ . The structure is stabilised by a three-

dimensional network of hydrogen bonds with all six available hydrogen atoms participating.

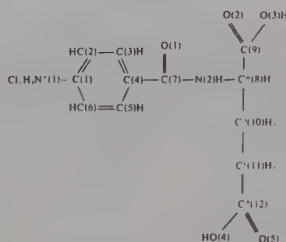


Fig. 1. The  $C_{12}H_{15}N_2O_5^+, Cl^-$  molecule.

t-BUTYLOXYCARBONYL-L-ALANYL-L-PROLINE MONOHYDRATE  
 $C_{13}H_{22}N_2O_5 \cdot H_2O$

M.E. KAMWAYA, O. OSTER, H. BRADACZEK, M.N. PONNUSWAMY, S. PARTHASARATHY, R. NAGARAJ and P. BALARAM, 1982. *Acta Cryst.*, **B38**, 172-176.

Orthorhombic,  $P2_12_12_1$ ,  $a = 20.751$ ,  $b = 13.457$ ,  $c = 5.875$  Å,  $D_m = 1.20$ ,  $Z = 4$ . Cu radiation,  $R = 0.039$  for 1664 reflexions.

There is one molecule of water as well as an N-H...O hydrogen bond (3.12 Å), rendering high stability to the crystal packing (Fig. 1). The water-bridge bond is of the same type as that of a triple helix:  $O_w...O$  2.78,  $O_w...O'$  2.69 and  $O_w...OH$  2.50 Å. Several five-membered ring systems, including pyrrolidine, are not planar. The  $C^\alpha$  atoms of the prolyl and alanyl residues are trans with respect to the peptide bond.

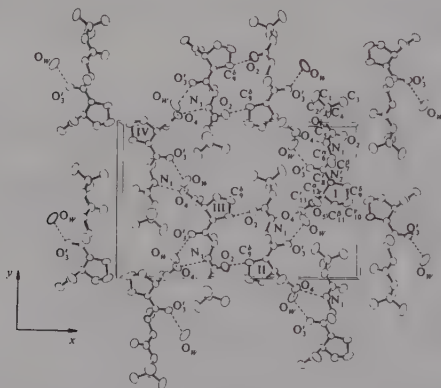


Fig. 1.  $C_{13}H_{22}N_2O_5 \cdot H_2O$ : projection of the crystal structure down  $c$ , hydrogen bonds are shown by broken lines.

N-ACETYLGLYCYL-L-LYSINE METHYL ESTER ACETATE  
 $C_{13}H_{25}N_3O_6$

D.M. SALUNKE and M. VIJAYAN, 1982. *Acta Cryst.*, **B38**, 287-289.

Orthorhombic,  $P2_12_12_1$ ,  $a = 5.511$ ,  $b = 14.588$ ,  $c = 21.109$  Å,  $Z = 4$ . Cu radiation,  $R = 0.079$  for 993 reflexions.

In the crystal structure (Fig. 1) the fully extended lysine side chain in the dipeptide molecule is staggered between the main-chain amino and carbonyl groups.

The dipeptide molecules are arranged in twofold helices centred on  $2_1$  screw axes and are linked by hydrogen bonds to one another ( $N...O$  2.87 and 2.76 Å) and to acetate ions ( $N...O$  2.88, 2.76 and 2.68 Å). Bond lengths and angles are normal.

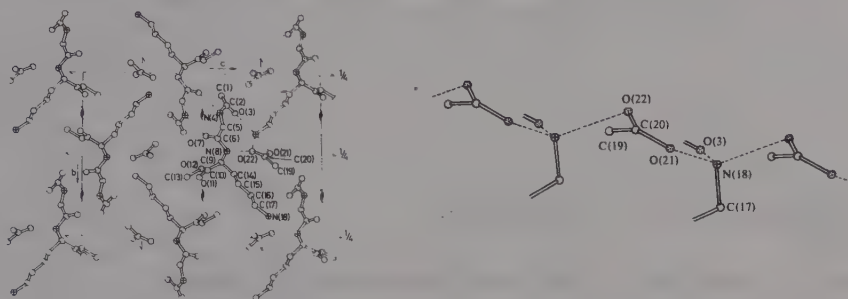


Fig. 1. Crystal structure of  $C_{13}H_{25}N_3O_6$  as viewed along the  $a$  axis, and the arrangement of the side-chain amino and acetate groups; broken lines indicate hydrogen bonds.

trans-2-HYDROXYCYCLOPENTYL p-BROMOBENZOYLGLYCINATE  
 $C_{14}H_{16}BrNO_4$

M. HAMELIN and Y. JEANNIN, 1982. Acta Cryst., B38, 1199-1203.

Monoclinic,  $C2/c$ ,  $a = 28.38$ ,  $b = 9.18$ ,  $c = 12.349$  Å,  $\beta = 108.76^\circ$ ,  $D_m = 1.49$ ,  $Z = 8$ .  
 Cu radiation,  $R = 0.060$  for 1655 reflexions.

In the crystal structure (Fig. 1) the molecules are linked into infinite chains by  $N-H...O$  and  $O-H...O$  hydrogen bonds, with  $N''...O(4)$  2.949 Å and  $O(4)...O(3')$  2.684 Å.

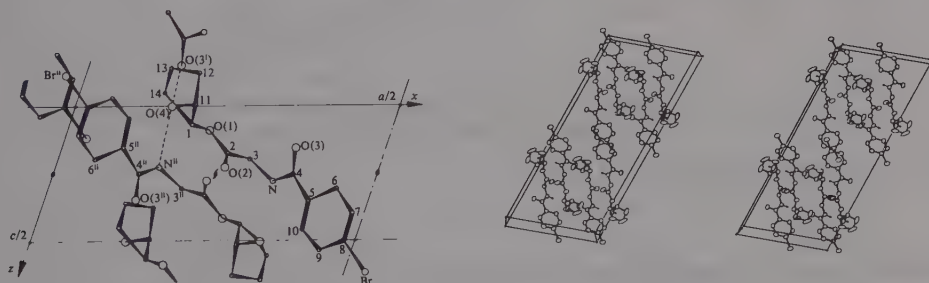


Fig. 1.  $C_{14}H_{16}BrNO_4$ : the structure as viewed down  $b$  (left) and a stereoscopic view of the structure (right).

PIVALOYL-L-PROLYL-N-METHYL-L-ALANINE METHYL ESTER  
 $C_{15}H_{26}N_2O_4$

A. AUBRY, B. VITOUX and M. MARRAUD, 1982. Cryst. Struct. Comm., 11, 135-140.

Orthorhombic,  $P2_12_12_1$ ,  $a = 6.776$ ,  $b = 13.412$ ,  $c = 18.588$  Å,  $Z = 4$ . Cu radiation,  
 $R = 0.049$  for 1104 reflexions.

The bond lengths and angles are within normal ranges with the exception of those related to thermally disordered C(7), C(8) and O(3) (Fig. 1) atoms. Both amide bonds and the ester function are in the trans conformation. All of them are approximately planar but both N(1) and N(2) nitrogens are slightly pyramidalized.



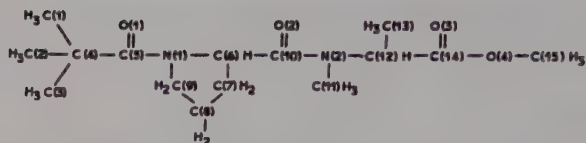
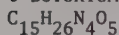


Fig. 1. The  $C_{15}H_{26}N_2O_4$  structure.

**t-BUTOXYCARBONYL-L-PROLYL-L-ALANYLGLYCINAMIDE**



T. KOJIMA, I. TANAKA and T. ASHIDA, 1982. *Acta Cryst.*, **B38**, 221-225.

Orthorhombic,  $P2_12_12_1$ ,  $a = 10.078$ ,  $b = 19.246$ ,  $c = 9.272$  Å,  $D_m = 1.25$ ,  $Z = 4$ . Cu radiation,  $R = 0.056$  for 2009 reflexions.

In the crystal structure (Fig. 1) the main chain of the peptide is folded at the Ala-Gly site into the  $\beta$ -turn type (I), in contrast with an extended conformation of the similar sequential peptide Poc-Pro-Ala-Gly-OH (1). The structure of this peptide has revealed that the widening of the  $NC^{\alpha}C^1$  angle of the second residue of the  $\beta$ -turn type (I) is highly significant for any kind of residue.

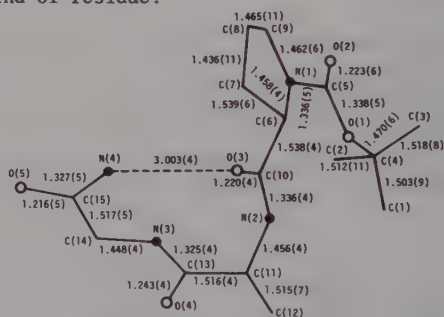
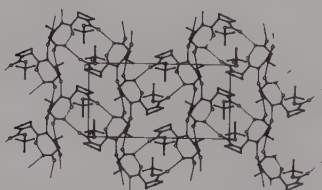
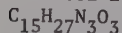


Fig. 1.  $C_{15}H_{26}N_4O_5$ : the crystal structure viewed along  $c$  with hydrogen bonds shown by thin lines, and bond lengths in the molecule.

1. Structure Reports, **48B**, 593.

**PIVALOYL-L-PROLYL- $\alpha$ -AMINOISOBUTYRYL-N-METHYLAMIDE**



B.V.V. PRASAD, H. BALARAM and P. BALARAM, 1982. *Biopolymers*, **21**, 1261-1273.

Monoclinic,  $P2_1$ ,  $a = 5.865$ ,  $b = 11.421$ ,  $c = 12.966$  Å,  $\beta = 97.55^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.061$  for 1250 reflexions.

The compound (Fig. 1) has a Type II  $\beta$ -turn conformation stabilized by a 4-1 hydrogen bond between methylamide N-H and pivaloyl CO group. The conformation angles are  $\phi$  Pro  $-57.8$ ,  $\psi$  Pro  $139.3$ ,  $\phi$  Aib  $61.4$ , and  $\psi$  Aib  $25.1^\circ$ .

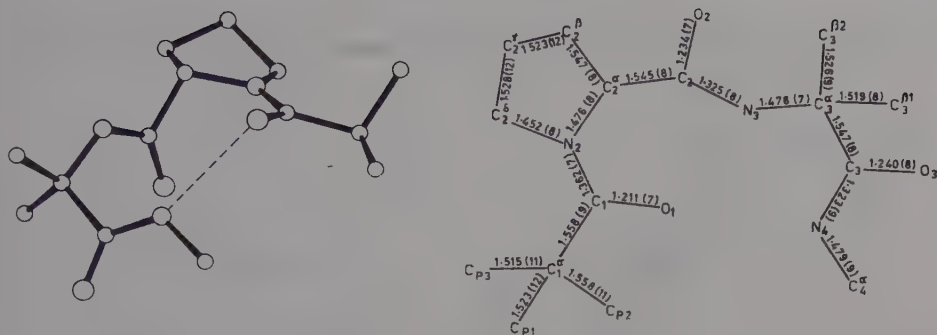
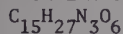


Fig. 1. The  $C_{15}H_{27}N_3O_3$  molecule and bond lengths.

METHYL N-t-BUTYLOXYCARBONYLGLYCYL-L-ALANYL- $\alpha$ -AMINOISOBUTYRATE



R. BOSCH, G. JUNG and W. WINTER, 1982. Justus Liebigs Ann. Chem., 1322-1329.

Orthorhombic,  $P2_12_12_1$ ,  $a = 10.043$ ,  $b = 11.590$ ,  $c = 16.779$  Å,  $Z = 4$ . Cu radiation,  $R = 0.0426$  for 1859 reflexions.

The molecule (Fig. 1) adopts a bent shape ( $\beta$ -turn III) due to an intramolecular hydrogen bond (N-H...O 3.087(5) Å). The remaining two N-H groups are involved in intermolecular hydrogen bonds (N-H...O 2.911(5), 2.943(5) Å). Bond lengths and angles have normal values.

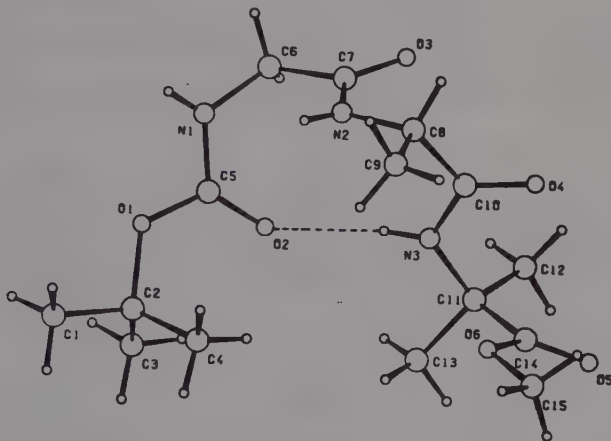


Fig. 1. The structure of  $C_{15}H_{27}N_3O_6$ .

## 2-N-ACETYLDEHYDROPHENYLALANYL-L-PROLINE HEMIHYDRATE



D. AJO, V. BUSETTI and G. GRANOZZI, 1982. *Tetrahedron*, **38**, 3329-3334.

Monoclinic, C2,  $a = 18.508$ ,  $b = 9.517$ ,  $c = 10.538$  Å,  $\beta = 120.00^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.048$  for 1078 reflexions.

The water molecule lies on a twofold axis and is linked to four peptides by O-H...O hydrogen bonding (2.762 and 2.709 Å). The conformation of the dehydro-Phe

residue (Fig. 1) is described by torsion angles  $\omega_1$  165.9,  $\phi_1$  -51.5,  $\psi_1$  135.2°. This conformation corresponds to one of six predicted energy minima.

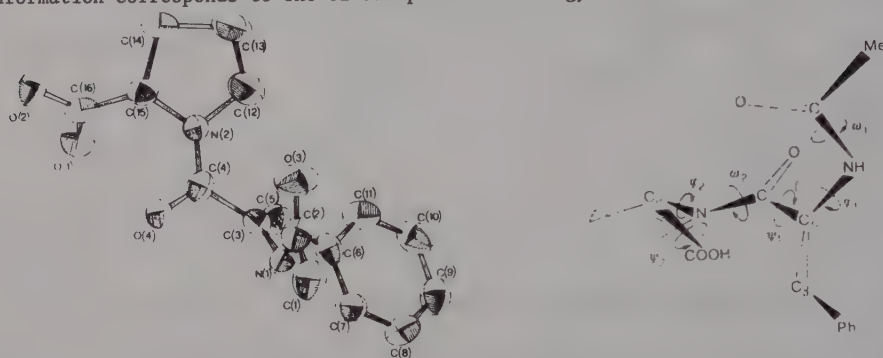
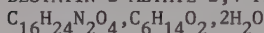


Fig. 1. The  $C_{16}H_{18}N_2O_4$  molecule and torsion angles.

# BESTATIN 2-METHYL-2,4-PENTANEDIOL DIHYDRATE



J.S. RICCI, Jr., A. BOUSVAROS and S. TAYLOR, 1982. *J. Org. Chem.*, **47**, 3063-3065.

Orthorhombic,  $P2_12_12_1$ ,  $a = 6.653$ ,  $b = 15.150$ ,  $c = 27.309$  Å,  $D_m = 1.18$ ,  $Z = 4$ . Cu radiation,  $R = 0.084$  for 2871 reflexions.

The structure (Fig. 1) indicates that the nonpolar phenylalanyl and leucyl side chains of bestatin are oppositely disposed in space and separated by  $\sim 10$  Å. The peptide bond is trans. It is unlikely that there is hydrogen bonding between the OH on C(8) and the adjacent carbonyl. On the assumption that the conformation of crystalline bestatin is similar to that which is found when it is at the active site, these data suggest possible modes of binding of this transition-state analogue to leucine aminopeptidase.

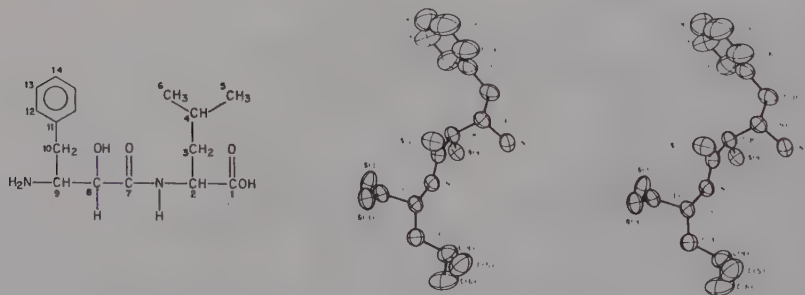
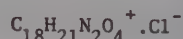
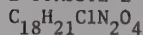


Fig. 1. The bestatin molecule and a stereoview.

# L-TYROSYL-L-PHENYLALANINE HYDROCHLORIDE



M. COTRAIT, Y. BARRANS and F. LEROY, 1982. *Acta Cryst.*, **B38**, 1626-1629.

Monoclinic,  $P2_1$ ,  $a = 10.432$ ,  $b = 10.188$ ,  $c = 8.561$  Å,  $\beta = 94.45^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.045$  for 1810 reflexions.

The dipeptide cation is hydrogen bonded to neighbouring cations and to  $Cl^-$  (Fig. 1) with  $O(16) \cdots O(12)$  2.592,  $Cl \cdots N(1)$  3.061,  $Cl \cdots O(8)$  3.157,  $O(8) \cdots N(1)$

3.058, O(17)...N(1) 2.878 Å. The peptide-NH and phenol O are not involved in hydrogen bonding. The molecule has an extended conformation with torsion angles Tyr:  $\psi$  153.6,  $\omega$  166.5,  $\psi_1$  -71.4,  $\psi_2$  112.7° and Phe:  $\phi$  -57.6,  $\psi$  135.7,  $\psi_1$  173.5,  $\psi_2$  83.3°. The N(1)-C(2) bond length is 1.502(5) Å.

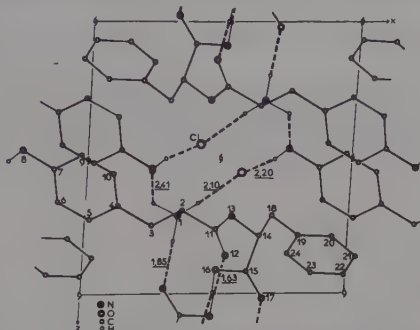


Fig. 1. The structure of L-tyrosyl-L-phenylalanine hydrochloride projected along *b*. Hydrogen bonds are shown by broken lines.

# BENZYLOXYCARBONYLPROLYLPROLINE

$C_{18}H_{22}N_2O_5$  (I)

# N-BENZYLOXYCARBONYLPROLYL-L-AZETIDINE-2-CARBOXYLIC ACID

$C_{17}H_{20}N_2O_5$  (II)

R.H. BLESSING and G.D. SMITH, 1982. *Acta Cryst.*, **B38**, 1203-1207.

I. Monoclinic,  $P2_1$ ,  $a = 10.446$ ,  $b = 14.527$ ,  $c = 6.107$  Å,  $\beta = 114.79^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.064$  for 1425 reflexions.

II. Monoclinic,  $P2_1$ ,  $a = 10.161$ ,  $b = 14.743$ ,  $c = 5.640$  Å,  $\beta = 109.63^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.048$  for 1702 reflexions.

The two crystals are isostructural. In each molecule (Fig. 1) the peptide bond is trans, but the urethane bond is cis, so that the benzoyloxycarbonyl group is folded back on the dipeptide. The dipeptide conformations are semi-extended. In each structure neighbouring molecules related to each other by the twofold screw axis are linked through hydrogen bonds from the carboxylic hydroxyl group of one molecule to the urethane carbonyl O atom of the next. The O...O distances are 2.648 (I) and 2.669 Å (II).

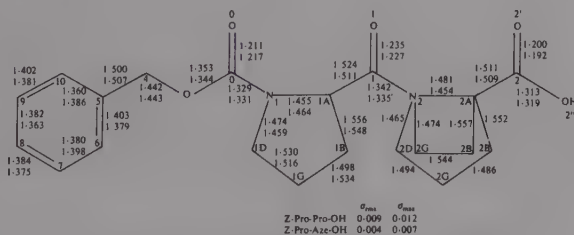


Fig. 1. Bond distances in  $C_{18}H_{22}N_2O_5$ (I) (upper values) and  $C_{17}H_{20}N_2O_5$  (II) (lower values).

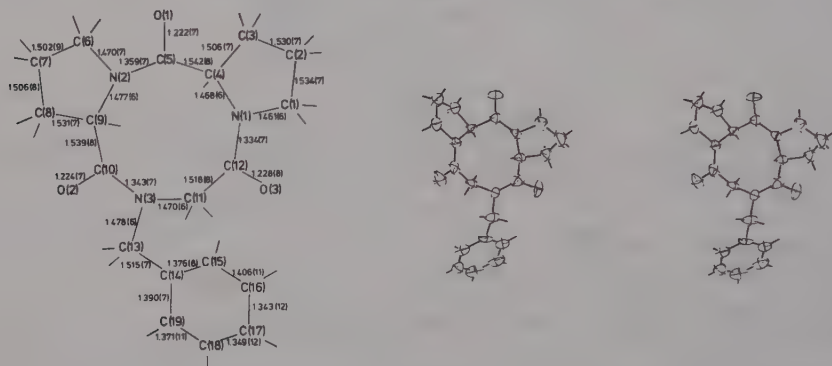
cyclo(-N-BENZYLGLYCYL-L-PROLYL-L-PROLYL-)

$C_{19}H_{23}N_3O_3$

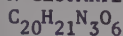
J.W. BATS and H. FUESS, 1982. Acta Cryst., B38, 1004-1007.

Orthorhombic,  $P2_12_12_1$ ,  $a = 5.688$ ,  $b = 11.782$ ,  $c = 25.727$  Å,  $Z = 4$ . Mo radiation,  $R = 0.056$  for 909 reflexions.

The molecule (Fig. 1) has a crown conformation with all three peptide bonds in the cis configuration. One of the peptide bonds differs significantly from planarity. The crystal packing shows two short intermolecular contacts between H and O atoms but no other contacts are shorter than the sum of the van der Waals radii of the atoms involved.



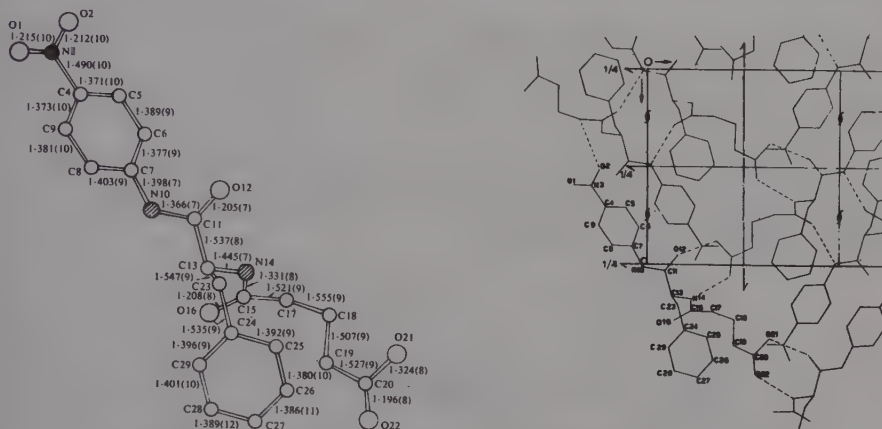
## N-GLUTARYL-L-PHENYLALANINE p-NITROANILIDE



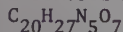
P. NARASIMHAN and V. PATTABHI, 1982. Acta Cryst., B38, 2293-2296.

Orthorhombic,  $P2_12_12_1$ ,  $a = 22.638$ ,  $b = 7.635$ ,  $c = 11.356$  Å,  $D_m = 1.352$ ,  $Z = 4$ . Co radiation,  $R = 0.053$  for 1258 reflexions.

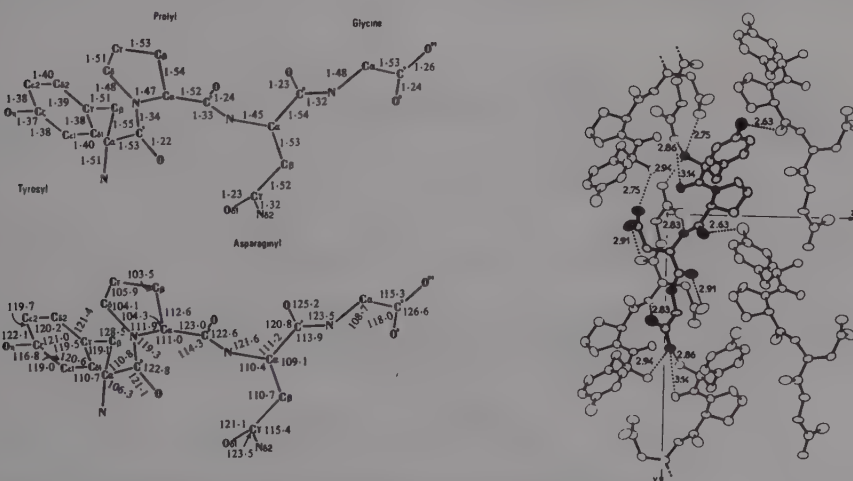
The molecules (Fig. 1) exist in the extended conformation and are linked in the crystal into a three-dimensional network by N-H...O (2.84 and 3.01 Å) and O-H...O (2.68 Å) hydrogen bonds.

Fig. 1.  $C_{20}H_{21}N_3O_6$ : bond lengths in the molecule and a packing diagram (projection down the b axis).

## L-TYROSYL-L-PROLYL-L-ASPARAGINYL-L-GLYCINE



G. PRÉCIGOUX, S. GEOFFRE, M. HOSPITAL and F. LEROY, 1982. Acta Cryst., B38, 2172-2176.

Fig. 1.  $C_{20}H_{27}N_5O_7$ : bond distances and angles in the molecule and the projection of the crystal packing in the plane  $yOz$ , showing hydrogen bonds.



Orthorhombic,  $P2_12_12_1$ ,  $a = 8.896$ ,  $b = 12.858$ ,  $c = 18.146$  Å,  $Z = 4$ . Cu radiation,  $R = 0.033$  for 1240 reflexions.

The molecule exists in the crystal as a zwitterion with extensive intermolecular O-H...O, N-H...O and O-H...N hydrogen bonding (2.63-3.14 Å) (Fig. 1). There is no intramolecular hydrogen bond and the peptide chain is in an extended configuration.

[N-(1-METHOXYCARBONYL-2-PHENYLETHYL)AZIRIDIN-2-YL]PHENYLALANINE METHYL ESTER  
 $C_{23}H_{26}N_2O_5$

A.F. MISHNEV, Ya.Ya. BLEIDELIS, A.V. EREMEEV, F.D. POLYAK and B.S. KATAEV, 1982. Zh. Strukt. Khim., 23-6, 86-90 [J. Struct. Chem., 23, 894-898].

Monoclinic,  $P2_1$ ,  $a = 5.182$ ,  $b = 23.491$ ,  $c = 9.109$  Å,  $\beta = 95.92^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.056$  for 1317 reflexions.

The molecule is shown in Fig. 1. The azylyl-phenylalanine dipeptide moiety showed a short C=O (1.21 Å) and a long C-N (1.35 Å) bond. The substituents of the three-membered ring have a trans configuration. The absolute configuration is established by that known for L-phenylalanine.

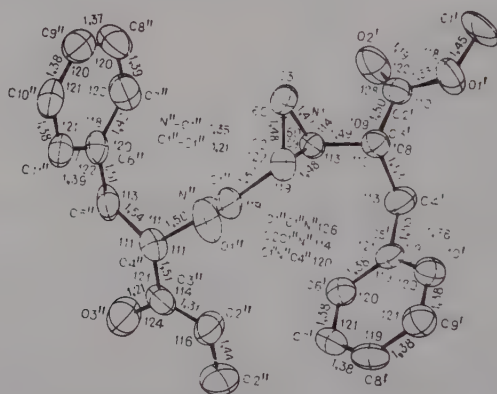


Fig. 1. The  $C_{23}H_{26}N_2O_5$  molecule.

POTASSIUM HYDROGEN BIS(2,4-DICHLOROPHENOXYISOBUTYRYLGLYCINATE)

$C_{24}H_{25}Cl_4KN_2O_8$

I. LEBAN, A. KOLBE, A. KOLBE and D. HADZI, 1982. J. Cryst. Spectrosc. Res., 12, 343-350.

Triclinic,  $P\bar{1}$ ,  $a = 6.071$ ,  $b = 7.891$ ,  $c = 15.622$  Å,  $\alpha = 84.57^\circ$ ,  $\beta = 80.57^\circ$ ,  $\gamma = 78.28^\circ$ ,  $D_m = 1.47$ ,  $Z = 1$ . Mo radiation,  $R = 0.082$  for 2772 reflexions.

The  $K^+$  and  $H^+$  ions occupy inversion centres and together with a short O...O distance of 2.430(9) Å (Fig. 1) classify the compound as a type A acid salt. The  $K^+$  ion has distorted octahedral coordination with  $K...O$  2.627-2.797(10) Å.

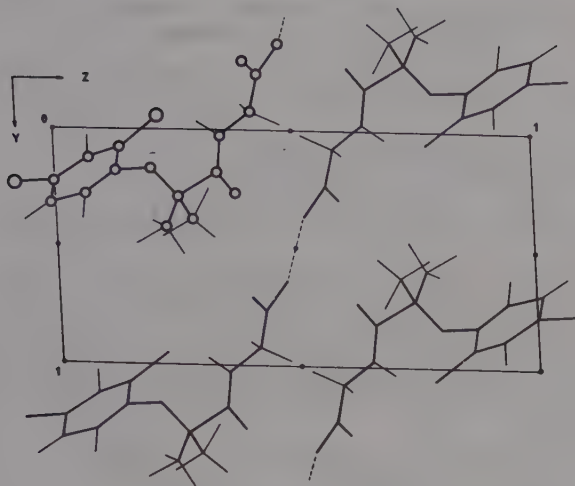


Fig. 1. A view of the crystal structure of  $C_{24}H_{25}Cl_4KN_2O_8$  along [100].

cyclo-(GLYCYL-L-PROLYL-L-PROLYLGLYCYL-L-PROLYL-L-PROLYL) TRIHYDRATE  
 $C_{24}H_{34}N_6O_6 \cdot 3H_2O$

M. CZUGLER, K. SASVARI and M. HOLLOSI, 1982. J. Am. Chem. Soc., 104, 4465-4469.

Orthorhombic,  $P2_12_12_1$ ,  $a = 9.237$ ,  $b = 13.972$ ,  $c = 20.851$  Å,  $Z = 4$ . Mo radiation,  $R = 0.034$  for 2298 reflexions.

The hexapeptide contains one transannular  $C=O \cdots HN$  hydrogen bond (Fig. 1). The 3  $H_2O$  molecules are involved in a coherent system of H-bonds which includes an intermolecular bifurcated H-bond, forming chains along  $b$ . One cis Gly-Pro and one cis-Pro-Pro linkage occur in consecutive positions. The most striking feature of the asymmetric conformation is the occurrence of a H-bonded type I  $\beta$ -turn encompassing two trans-configured proline residues in the other half of the molecule.

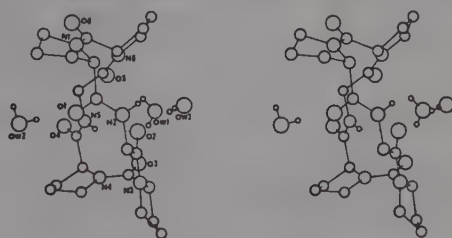


Fig. 1. Stereoview of  $C_{24}H_{34}N_6O_6 \cdot 3H_2O$ .

N-BENZYL-OXYCARBONYL-TRIS(2-METHYLALANYL)-*t*-BUTYL ESTER  
 $C_{24}H_{37}N_3O_6$  (I)

N-BENZYL-OXYCARBONYL-TRIS(2-METHYLALANYL)-2-METHYLALANINE  
 $C_{24}H_{36}N_4O_7$  (II)

N-BENZYL-OXYCARBONYL-PENTAKIS(2-METHYLALANYL)-*t*-BUTYL ESTER  
 $C_{32}H_{51}N_5O_8$  (III)

E. BENEDETTI, A. BAVOSO, B. Di BLASIO, V. PAVONE, C. PEDONE, M. CRISMA, G.M. BONORA and C. TONIOLO, 1982. *J. Am. Chem. Soc.*, **104**, 2437-2444.

I. Monoclinic,  $P2_1/c$ ,  $a = 9.540$ ,  $b = 26.026$ ,  $c = 11.006$  Å,  $\beta = 106.72^\circ$ ,  $D_m = 1.17$ ,  $Z = 4$ . Cu radiation,  $R = 0.053$  for 2443 reflexions.

II. Monoclinic,  $P2_1/n$ ,  $a = 16.678$ ,  $b = 17.891$ ,  $c = 9.016$  Å,  $\beta = 95.92^\circ$ ,  $D_m = 1.22$ ,  $Z = 4$ . Cu radiation,  $R = 0.075$  for 4189 reflexions.

III. Triclinic,  $P\bar{1}$ ,  $a = 9.185$ ,  $b = 11.540$ ,  $c = 18.737$  Å,  $\alpha = 105.79$ ,  $\beta = 93.92$ ,  $\gamma = 102.98^\circ$ ,  $D_m = 1.13$ ,  $Z = 2$ . Mo radiation,  $R = 0.062$  for 3734 reflexions.

The structure analyses of the N-benzoyloxycarbonyl homotri-, tetra-, and pentapeptides from  $\alpha$ -aminoisobutyric acid (I-III respectively, see Fig. 1) have shown the occurrence of incipient  $3_{10}$  helices, formed by one, two, and three type III (or type III')  $\beta$  turns, respectively.  $\alpha$ -Helical structures, although having closely related pairs of  $\phi, \psi$  torsional angles, are not compatible with the observed intramolecular N-H...O=C hydrogen-bonding schemes of the tetra- and pentapeptides. Bond lengths are normal.

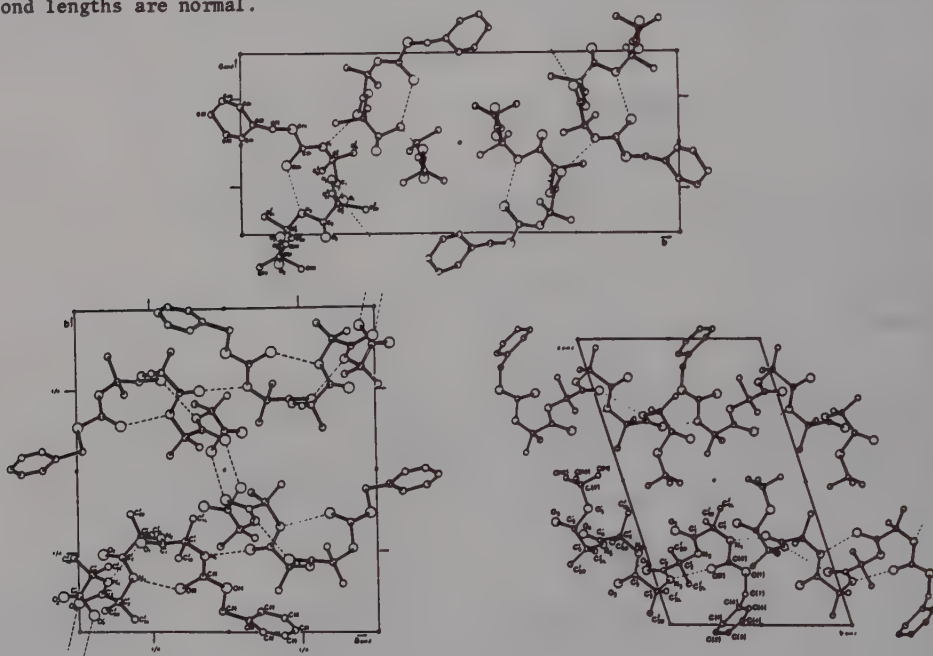


Fig. 1. The structures of  $C_{24}H_{37}N_3O_6$  (top),  $C_{24}H_{36}N_4O_7$  (lower left), and  $C_{32}H_{51}N_5O_8$  (lower right).

N-(PHENYLACETYL-L-ALANYL)CYCLO(L-PHENYLALANYL-D-PROLYL)  
 $C_{25}H_{27}N_3O_4$

G. LUCENTE, F. PINNEN, G. ZANOTTI, S. CERRINI, W. FEDELI and F. MAZZA, 1982. *Cryst. Struct. Comm.*, **11**, 1487-1493.

Monoclinic,  $P2_1$ ,  $a = 9.956$ ,  $b = 8.809$ ,  $c = 13.615$  Å,  $\beta = 111.0^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.037$  for 2500 reflexions.

The diketopiperazine ring (Fig. 1) has a boat conformation and the pyrrolidine ring is a  $\beta$ -envelope. The phenylalanine side chain is axial.

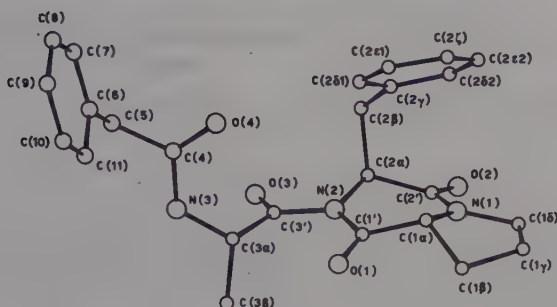


Fig. 1. A general view of  $C_{25}H_{27}N_3O_4$ .

METHYL N-p-CHLOROBENZOYL-L-PROLYL-2-METHYLALANYL-L-ALANYL-2-METHYLALANYL-L-ALANINATE  
 $C_{27}H_{38}ClN_5O_7$

T.S. CAMERON, A.W. HANSON and A. TAYLOR, 1982. Cryst. Struct. Comm., **11**, 321-330.

Triclinic,  $P1$ ,  $a = 9.977$ ,  $b = 10.210$ ,  $c = 8.828$  Å,  $\alpha = 90.68$ ,  $\beta = 106.44$ ,  $\gamma = 63.82^\circ$ ,  $D_m = 1.254$ ,  $Z = 1$ . Cu radiation,  $R = 0.036$  for 2341 reflexions.

The conformation of the peptide is shown in Fig. 1. The torsion angles and the H-bonding network are indicative of a 3-residue segment (-Aib-L-Ala-Aib-) of left-handed  $3_10$  helix. The bond angles about the  $\alpha$  carbon atoms of the two Aib residues demonstrate the typical asymmetry of Aib residues in left-handed helices:  $\tau(NC^\alpha C_L^\beta)$  and  $\tau(C_L^\beta C^\alpha C')$  are greater than the tetrahedral value of  $109.45^\circ$ , while  $\tau(NC^\alpha C_B^\beta)$  and  $\tau(C_B^\beta C^\alpha C')$  are less than the tetrahedral value. The intermolecular H-bond joins molecules related by the  $b$  translation. The structure thus consists of endless chains along the  $b$ -axis.

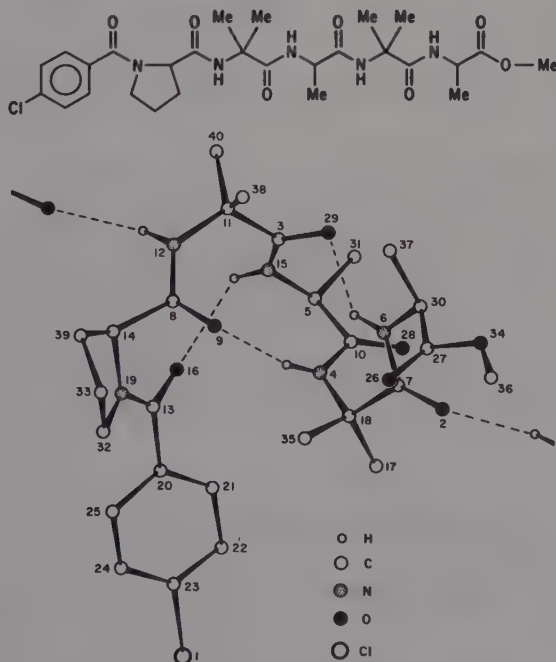


Fig. 1. The  $C_{27}H_{38}ClN_5O_7$  molecule.

cyclo-(O-BENZYL-L-THREONYL-D-VALYL-L-PROLYLSARCOSYL-N-METHYL-L-ALANYL)  
 $C_{28}H_{41}N_5O_6$

A.B. MAUGER, O.A. STUART, R.J. HIGHET and J.V. SILVERTON, 1982. J. Am. Chem. Soc., 104, 174-180.

Monoclinic,  $P2_1$ ,  $a = 10.167$ ,  $b = 28.047$ ,  $c = 10.581$  Å,  $\beta = 106.39^\circ$ ,  $D_m = 1.26$ ,  $Z = 4$ . Cu radiation,  $R = 0.037$  for 4992 reflexions.

The two crystallographically independent molecules of the cyclic pentapeptide are shown in Fig. 1. Important structural features include trans peptide bonds, a Sar-MeAla  $\beta$  turn, and a possible (long) 4 $\rightarrow$ 1 (Thr-Pro) hydrogen bond. Bond lengths ( $\sigma = 0.004$ - $0.006$  Å) are as expected.

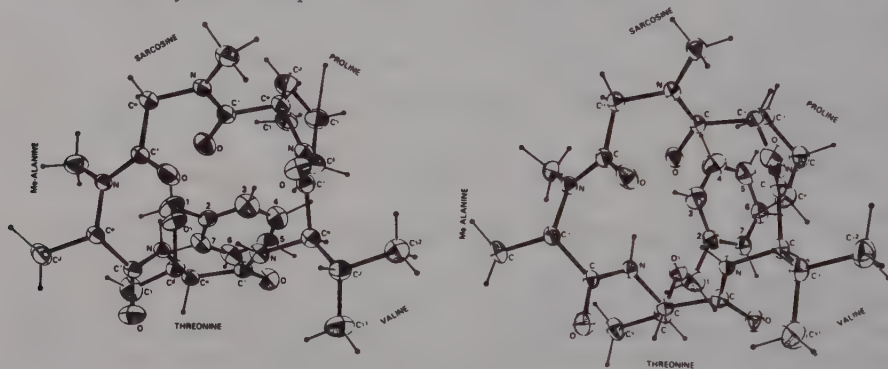


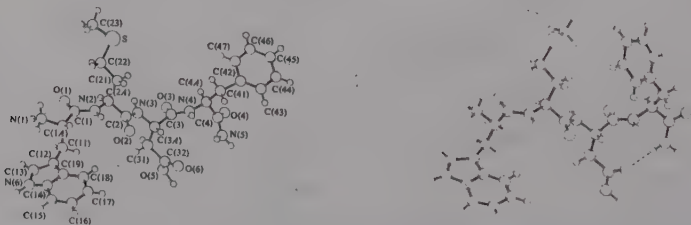
Fig. 1. The two independent molecules of  $C_{28}H_{41}N_5O_6$ .

L-TRYPTOPHANYL-L-METHIONYL-L-ASPARTYL-L-PHENYLALANYLAMIDE HYDROCHLORIDE METHANOL  
 HEMIETHER SOLVATE  
 $C_{29}H_{37}ClN_6O_6S, CH_4O, 0.5(C_4H_{10}O)$

W.B.T. CRUSE, E. EGERT, M.A. VISWAMITRA and O. KENNARD, 1982. Acta Cryst., B38, 1758-1764.

Monoclinic,  $P2_1$ ,  $a = 14.391$ ,  $b = 23.757$ ,  $c = 11.362$  Å,  $\beta = 103.48^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.082$  for 3143 reflexions.

The two peptide molecules in the asymmetric unit have different conformations (Fig. 1); one is extended while the other has the phenyl group bent back towards the backbone with an intramolecular hydrogen bond between the terminal amide group and the Asp side chain. The hydrogen-bonded peptide dimer is the repeat unit of an infinite, antiparallel,  $\beta$ -pleated sheet along  $c$ . These sheets are connected by a dense network of hydrogen bonds involving  $Cl^-$  ions. Packing of the side chains is stabilised by both solvent components;  $CH_3OH$  accepts hydrogen bonds from Trp and Asp while the ether molecules fill large pockets enclosed by the hydrophobic peptide residues.





t-BUTOXYCARBONYL- $\alpha$ -AMINOISOBUTYRYL-L-PROLYL-L-VALYL- $\alpha$ -AMINOISOBUTYRYL-L-VALINE METHYL  
ESTER MONOHYDRATE  
 $C_{29}H_{51}N_5O_8 \cdot H_2O$

A.K. FRANCIS, M. IQBAL, P. BALARAM and M. VIJAYAN, 1982. J. Chem. Soc. Perkin II, 1235-1239.

Orthorhombic,  $P2_12_12_1$ ,  $a = 10.192$ ,  $b = 10.440$ ,  $c = 32.959$  Å,  $D_m = 1.158$ ,  $Z = 4$ . Cu radiation,  $R = 0.101$  for 1827 reflexions.

The bond length deviations from expected values in this protected amino-terminal pentapeptide from suzukacillin can be accounted for by the highly anisotropic thermal parameters. The expanded bond angles  $C(9)-C(12)-N(14)$  and  $C(12)-N(14)-C(15)$  (Fig. 1) result from steric interactions between the side chains of the prolyl and amino-isobutyryl groups. The chain conformation is a right handed fourfold helix with a pitch of 5.58 Å. There are three N-H...O hydrogen bonds, two 5+1 types and one 4+1 type, involving three separate amide nitrogens as donors, but only two carbonyl oxygen acceptors, with that of the protecting group accepting two hydrogen bonds. The prolyl carbonyl forms a hydrogen bond to the water molecule which is also hydrogen bonded to the carbonyl of the non-terminal valyl residue of a symmetry related molecule. The helix axis of the molecule lies at  $22^\circ$  to the b axis with the molecules stacked head-to-tail roughly parallel to the helix axis. Each column is bridged through the water molecules to a column related by a  $2_1$  screw axis parallel to b.

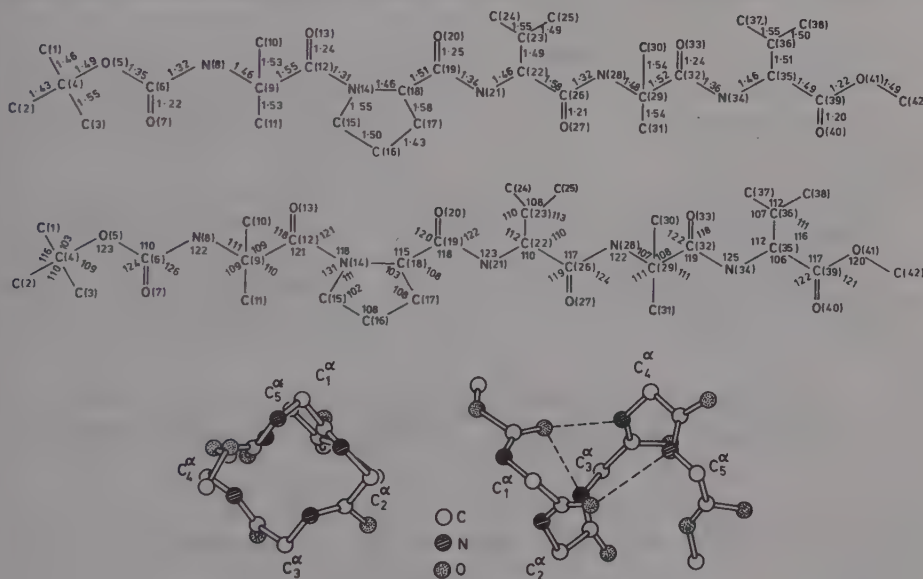


Fig. 1. Bond lengths and angles in  $C_{29}H_{51}N_5O_8 \cdot H_2O$  (upper) and the molecular conformation viewed along the helix axis (lower left) and parallel to it (lower right).

N-(t-BUTOXYCARBONYL)-L-PHENYLALANYL-L-PHENYLALANINE BENZYL ESTER  
 $C_{30}H_{34}N_2O_5$  (I)

L-PHENYLALANYL-L-PHENYLALANINE ETHYL ESTER TRIFLUOROACETATE  
 $C_{22}H_{25}F_3N_2O_5$  (II)

$C_{20}H_{25}N_2O_3^+ \cdot C_2F_3O_2^-$

O. YAMASHITA, Y. KATO, T. YAMANE and T. ASHIDA, 1982. Acta Cryst., B38, 2657-2663.



I. Monoclinic,  $P2_1$ ,  $a = 14.363$ ,  $b = 19.016$ ,  $c = 5.077$  Å,  $\beta = 97.02^\circ$ ,  $D_m = 1.206$ ,  $Z = 2$ . Cu radiation,  $R = 0.070$  for 1921 reflexions.

II. Orthorhombic,  $P2_12_12_1$ ,  $a = 16.507$ ,  $b = 24.927$ ,  $c = 5.650$  Å,  $D_m = 1.283$ ,  $Z = 4$ . Cu radiation,  $R = 0.074$  for 1544 reflexions.

The main chain of peptide I has an extended conformation (Fig. 1). The molecules are linked in the crystal by N-H...O hydrogen bonds with N...O 2.960(5) and 3.143(5) Å. Peptide II has a column structure with a hydrophilic core of hydrogen bonds and a hydrophobic shell (Fig. 1). The molecules are linked in the crystal by N-H...O hydrogen bonds with N...O 2.820(10), 2.962(10), 2.726(10) and 2.924(8) Å.

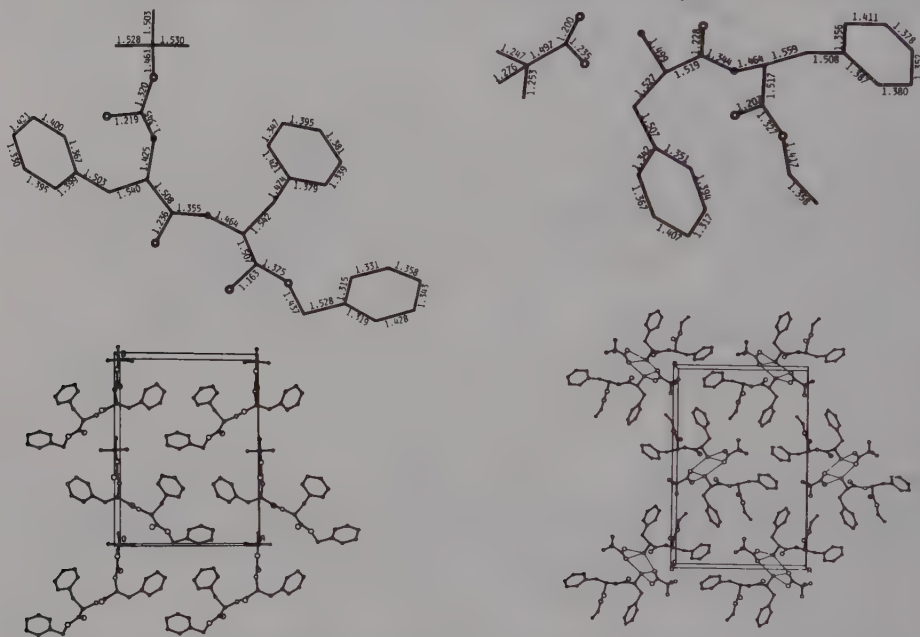


Fig. 1.  $C_{30}H_{34}N_2O_5$  (I): bond lengths (upper left) and a view of the crystal structure (lower left);  $C_{20}H_{25}N_2O_3^+$ ,  $C_2F_3O_2^-$  (II) bond lengths (upper right) and a view of the crystal structure (lower right).

t-BUTYLOXYCARBONYL-LEUCYL- $\alpha$ -AMINOISOBUTYRYL-PROLYL-VALYL- $\alpha$ -AMINOISOBUTYRIC ACID METHYL ESTER

$C_{30}H_{53}N_5O_8$

C.P. RAO and P. BALARAM, 1982. Biopolymers, **21**, 2461-2472.

Monoclinic,  $P2_1$ ,  $a = 11.034$ ,  $b = 10.894$ ,  $c = 15.483$  Å,  $\beta = 104.80^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.069$  for 2546 reflexions.

A perspective view of the molecule is shown in Fig. 1. The pentapeptide folds into a right-handed  $3_{10}$  helical conformation, stabilized by two intramolecular 4+1 hydrogen bonds between the Val(4) NH and Leu(1) CO (N5...O2 3.154 Å) and Aib(5) NH and Aib(2) CO (N6...O3 3.007 Å) groups. All the peptide bonds in the molecule are nearly planar. Bond lengths and angles are largely unexceptional.

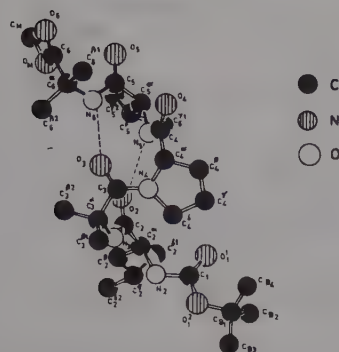


Fig. 1. A view of the  $C_{30}H_{53}N_5O_8$  molecule.

CYCLO(L-METHYLVALYL-D-HYDROXYISOVALERYL-L-METHYLVALYL-L-HYDROXYISOVALERYL-D-METHYLVALYL-L-HYDROXYISOVALERYL)

$C_{33}H_{57}N_3O_9$

N.E. ZHUKHLISTOVA, G.N. TISHCHENKO and K.M. POLYAKOV, 1982. Kristallografiya, 27, 288-295 [Sov. Phys. Crystallogr., 27, 176-181].

Monoclinic,  $P2_1$ ,  $a = 13.516$ ,  $b = 6.049$ ,  $c = 23.332$  Å,  $\gamma = 101.88^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.052$  for 2086 reflexions.

The molecule of the LDLLDL analogue of enniatin B (Fig. 1) has an asymmetric conformation. Three carbonyl groups (those of the 2nd, 3rd and 5th residues) are directed downward, and two (those of the 4th and 6th residues) upward from the median plane of the ring which has a slightly elongated shape. The CO group of the first residue lies approximately in the plane of the ring and points inward. The side radical of one residue statistically occupies two positions, one with trans and the other with gauche configurations of the fragment  $C^{\alpha}H-C^{\beta}H$ . Mean bond lengths are:  $N_j-C_j^{\alpha}$  1.476,  $N_j-C_j^{\beta}$  1.341, and  $N_j-C_j$  1.460 Å.

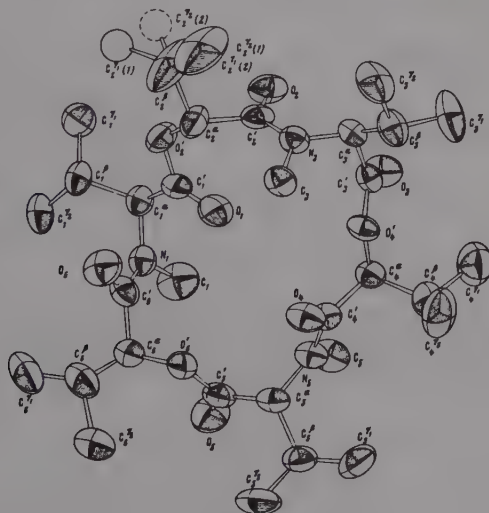


Fig. 1. Molecular structure of  $C_{33}H_{57}N_3O_9$ .

CYCLO(-L-PHENYLALANINE-D-LEUCINE-GLYCINE-L-PHENYLALANINE-L-LEUCINE-GLYCINE-)  
TETRAHYDRATE  
 $C_{34}H_{46}N_6O_6 \cdot 4H_2O$  (I)

CYCLO(-L-PHENYLALANINE-D-LEUCINE-GLYCINE-D-PHENYLALANINE-L-LEUCINE-GLYCINE-) DIHYDRATE  
 $C_{34}H_{46}N_6O_6 \cdot 2H_2O$  (II)

C.L. BARNES and D. VAN DER HELM, 1982. *Acta Cryst.*, **B38**, 2589-2595.

I. Monoclinic,  $P2_1$ ,  $a = 7.056$ ,  $b = 36.58$ ,  $c = 7.551$  Å,  $\beta = 104.12^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.0835$  for 3595 reflexions (at 138 K).

II. Triclinic,  $P\bar{1}$ ,  $a = 8.3489$ ,  $b = 16.634$ ,  $c = 6.848$  Å,  $\alpha = 100.997$ ,  $\beta = 108.911$ ,  $\gamma = 88.03^\circ$ ,  $Z = 1$ . Cu radiation,  $R = 0.0459$  for 3626 reflexions (at 138 K).

Both peptides exhibit the common conformation of two  $\beta$ -turns linked by extended glycol residues, but only one of the two possible transannular hydrogen bonds is present in I, while in II no transannular hydrogen bonds are observed (Fig. 1). Bond lengths and angles compare well in the two structures and with those in other cyclic hexapeptides.

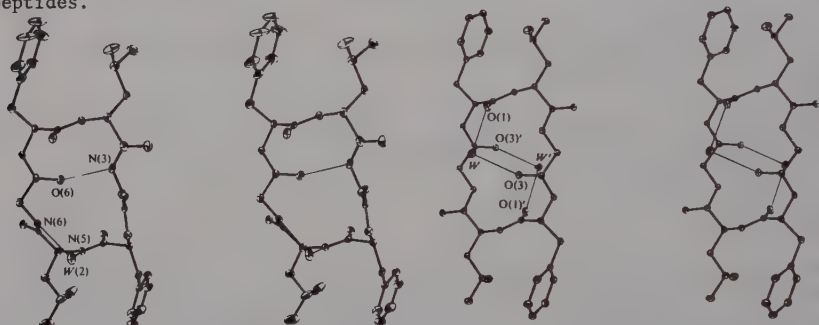


Fig. 1. Stereoviews of  $C_{34}H_{46}N_6O_6 \cdot 4H_2O$  (I) and  $C_{34}H_{46}N_6O_6 \cdot 2H_2O$  (II).

(5,10,15,20-TETRAMETHYLPORPHYRINATO)NICKEL(II)  
 $C_{24}H_{20}N_4Ni$  (I)

(5,10,15,20-TETRAMETHYLCHLORINATO)NICKEL(II)  
 $C_{24}H_{22}N_4Ni$  (II)

J.C. GALLUCCI, P.N. SWEPSTON and J.A. IBERS, 1982. *Acta Cryst.*, **B38**, 2134-2139.

I. Monoclinic,  $P2_1/c$ ,  $a = 11.332$ ,  $b = 5.664$ ,  $c = 14.162$  Å,  $\beta = 104.80^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.042$  for 4438 reflexions (at 140 K).

II. Monoclinic,  $P2_1/n$ ,  $a = 12.593$ ,  $b = 7.489$ ,  $c = 19.296$  Å,  $\beta = 94.08^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.043$  for 6602 reflexions (at 123 K).

The two complexes (Fig. 1) have markedly different structures: the porphyrin complex (I) is nearly planar whilst the chlorin complex (II) has an  $S_4$ -ruffled conformation. The asymmetry in chemically equivalent bonds in II indicates a reduction in aromaticity relative to I.

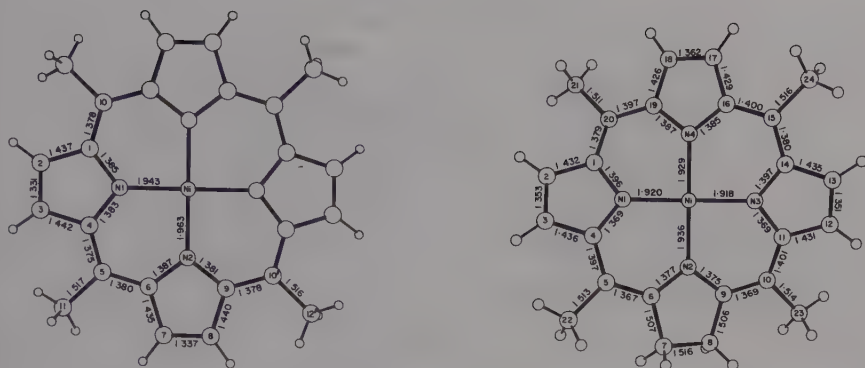


Fig. 1. Bond distances in  $C_{24}H_{20}N_4Ni$  (I) (left) and  $C_{24}H_{22}N_4Ni$  (II) (right).

2,2,8,8,12,13,17,18-OCTAMETHYLISOBACTERIOCHLORIN  
 $C_{28}H_{34}N_4$

W.B.T. CRUSE, P.J. HARRISON and O. KENNARD, 1982. J. Am. Chem. Soc., 104, 2376-2380.

Rhombohedral,  $R\bar{3}$ ,  $a = 16.926 \text{ \AA}$ ,  $\alpha = 109.72^\circ$  (hexagonal cell has  $a = 27.682$ ,  $c = 16.717 \text{ \AA}$ ),  $Z = 18$ . Cu radiation,  $R = 0.049$  for 2205 reflexions.

The structure (Fig. 1) is a superposition of two tautomeric forms in which diagonally opposed nitrogen atoms have bonded H atoms. The macrocycle is planar in contrast to metalated isobacteriochlorins which are generally domed or buckled.

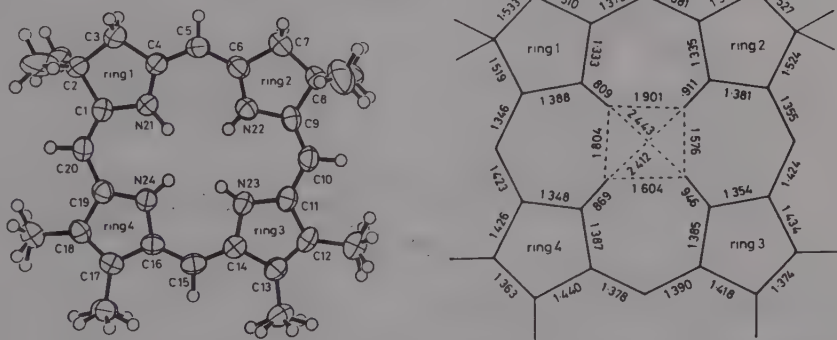


Fig. 1. The structure of  $C_{28}H_{34}N_4$ , selected bond lengths ( $\sigma = 0.005\text{-}0.007 \text{ \AA}$ ) at right.

2,2,7,7,12,12,17,17-OCTAMETHYL-2,3,4,7,8,12,13,18,19,20-DECAHYDRO-17H-PORPHYRINATO-NICKEL(II) PERCHLORATE METHYL ACETATE  
 $C_{28}H_{39}ClN_4NiO_4 \cdot C_3H_6O_2$

A. FASSLER, A. PFALTZ, P.M. MULLER, S. FAROOQ, C. KRATKY, B. KRAUTLER and E. ESCHENMOSER, 1982. Helv. Chim. Acta, 65, 812-827.

Monoclinic,  $P2_1/c$ ,  $a = 10.827$ ,  $b = 10.559$ ,  $c = 28.83 \text{ \AA}$ ,  $\beta = 96.40^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.163$  for 1259 reflexions.

The analysis establishes the structure as shown in Fig. 1.

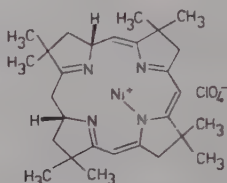
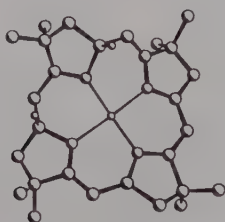


Fig. 1. The  $C_{28}H_{39}ClNbNiO_4$  molecule.

# DICHLORO(PHTHALOCYANINATO)NIOBIUM(IV)

$C_{32}H_{16}Cl_2N_8Nb$

$[NbCl_2(C_{32}H_{16}N_8)]$

K. UKEI, 1982. Acta Cryst., B38, 1288-1290.

Monoclinic,  $P2_1/c$ ,  $a = 12.557$ ,  $b = 10.315$ ,  $c = 20.571$  Å,  $\beta = 90.59^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.042$  for 4049 reflexions.

In the complex (Fig. 1) Nb is coordinated to four isoindole N atoms and two Cl atoms (Nb-N 2.125, 2.162, 2.140, 2.148(4), Nb-Cl 2.403 and 2.409(2) Å), and is displaced 0.98 Å from the central  $C_8N_8$  plane.

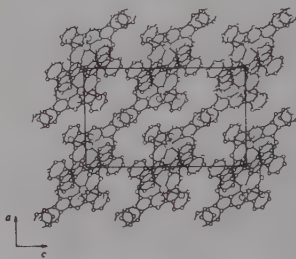
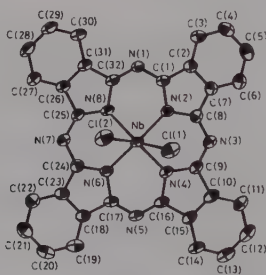


Fig. 1. Dichloro(phthalocyaninato)niobium(IV): a perspective view of the complex molecule and the unit cell viewed along the  $b$  axis.

# OXOPHTHALOCYANINATOTITANIUM(IV) (Two Phases)

$C_{32}H_{16}N_8OTi$

W. HILLER, J. STRAHLE, W. KOBEL and M. HANACK, 1982. Z. Krist., 159, 173-183.

I. Monoclinic,  $P2_1/c$ ,  $a = 13.411$ ,  $b = 13.230$ ,  $c = 13.810$  Å,  $\beta = 103.72^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.072$  for 1794 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 12.166$ ,  $b = 12.584$ ,  $c = 8.641$  Å,  $\alpha = 96.28^\circ$ ,  $\beta = 95.03^\circ$ ,  $\gamma = 67.86^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.072$  for 2054 reflexions.

In both modifications the Ti atom has square-pyramidal coordination (Fig. 1) with Ti-O 1.65 and 1.63 Å in I and II respectively. The Ti-N distances are in the range 2.043(8) to 2.084(4) Å with a mean value 2.07 Å.

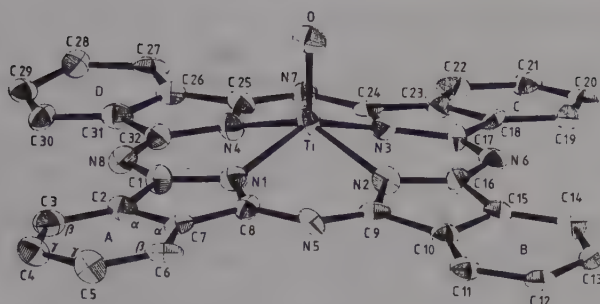


Fig. 1. The monoclinic form of  $C_{32}H_{16}N_8OTi$ .

# LEAD PHTHALOCYANINE

$C_{32}H_{16}N_8Pb$

Y. IYECIKA, K. YAKUSHI, I. IKEMOTO and H. KURODA, 1982. *Acta Cryst.*, **B38**, 766-770.

Triclinic,  $P\bar{1}$ ,  $a = 13.123$ ,  $b = 16.131$ ,  $c = 12.889$  Å,  $\alpha = 94.22$ ,  $\beta = 96.20$ ,  $\gamma = 114.19^\circ$ ,  $D_m = 1.93$ ,  $Z = 4$ . Mo radiation,  $R = 0.059$  for 7683 reflexions.

The structure of the monoclinic form of lead phthalocyanine has already been reported (1). In the triclinic crystal two independent molecular columns exist but they have almost equivalent structures (Fig. 1). The molecules are considerably distorted from  $C_{4v}$  symmetry. The central Pb atom significantly deviates from the convex side of the phthalocyanine skeleton, and this deviation is larger than that in the monoclinic form. Molecules are stacked along the  $a$  axis and oriented so that their convex sides alternate. They are considerably inclined with respect to the stacking axis.

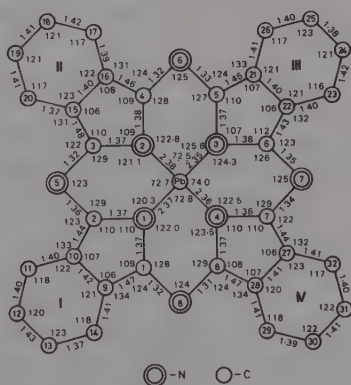
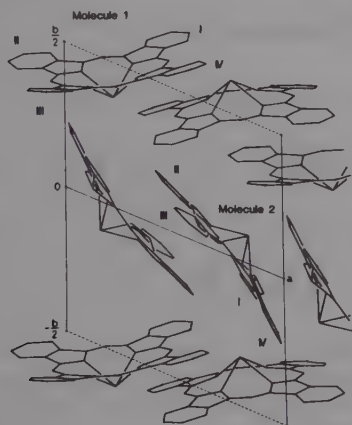


Fig. 1.  $C_{32}H_{16}N_8Pb$ : projection of the crystal structure on to the (001) plane (left) and average values of the bond lengths and angles in molecules 1 and 2 (right).

1. *Structure Reports*, **39B**, 365.



## (TETRABENZOPORPHYRINATO)NICKEL(II) IODIDE

 $C_{36}H_{20}IN_4Ni$  $Ni(C_{36}H_{20}N_4)I$ 

J. MARTINSEN, L.J. PACE, T.E. PHILLIPS, B.M. HOFFMAN and J.A. IBERS, 1982. J. Am. Chem. Soc., 104, 83-91.

Tetragonal,  $P4/mcc$ ,  $a = 14.081$ ,  $c = 6.434 \text{ \AA}$ ,  $D_m = 1.72$ ,  $Z = 2$ . Mo radiation,  $R = 0.055$  for 330 reflexions. (Cell constants, structure at 113 K,  $D_m$  at 298 K.)

The structure consists of planar  $Ni(C_{36}H_{20}N_4)$  units (Fig. 1) stacked metal-over-metal with a  $Ni...Ni$  spacing of  $3.217(5) \text{ \AA}$  at 113 K and with the units staggered by  $41^\circ$ . Running parallel to these stacks are chains of disordered  $I_3^-$  ions. Their existence requires a mixed valency (partially oxidized) formulation:  $[Ni(C_{36}H_{20}N_4)]^{0.33+}(I_3^-)_{1/3}$ . The unique Ni-N distance is  $1.966(9) \text{ \AA}$ .

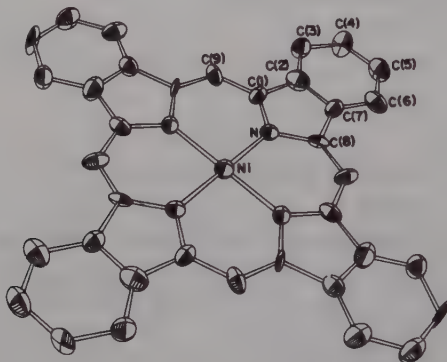


Fig. 1. The  $Ni(C_{36}H_{20}N_4)$  molecule.

## FLUORO(2,3,7,8,12,13,17,18-OCTAETHYLPORPHYRINATO)OXONIUMIUM

 $C_{36}H_{44}FNbO$ 

G. LECOMTE, J. PROTAS, P. RICHARD, J.M. BARBE and R. GUILARD, 1982. J. Chem. Soc. Dalton, 247-250.

Monoclinic,  $P2_1/c$ ,  $a = 15.007$ ,  $b = 22.257$ ,  $c = 10.058 \text{ \AA}$ ,  $\beta = 77.85^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.050$  for 5551 reflexions.

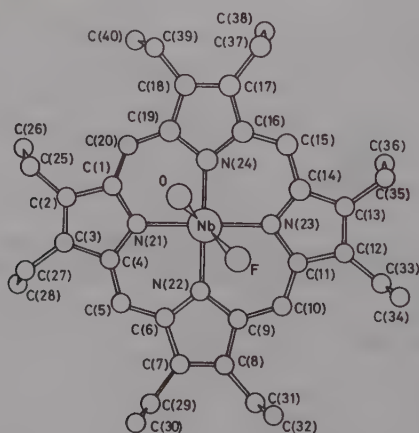
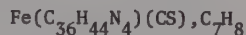


Fig. 1.  $C_{36}H_{44}FNbO$ : the molecular structure.

The Nb atom of the molecule (Fig. 1) exhibits a cis geometry of the 4:2 type (Nb-O 1.749(3), Nb-F 1.888(3), Nb-N (mean) 2.210(3) Å). The Nb atom lies 0.906(3) Å from the perfect 4N equatorial plane. The porphyrin ring is domed and the O and F ligands eclipse C(10) and C(20) of the porphyrin. The O-Nb-F angle is 89.0(1)°.

(THIOCARBONYL) (OCTAETHYLPORPHINATO) IRON(II) TOLUENE



W.R. SCHEIDT and D.K. GEIGER, 1982. *Inorg. Chem.*, 21, 1208-1211.

Triclinic,  $\bar{P}1$ ,  $a = 12.396$ ,  $b = 15.393$ ,  $c = 10.726$  Å,  $\alpha = 93.08$ ,  $\beta = 98.62$ ,  $\gamma = 75.53^\circ$ ,  $D_m = 1.26$ ,  $Z = 2$ . Mo radiation,  $R = 0.070$  for 6919 reflexions.

The molecule (Fig. 1) displays distorted square pyramidal coordination about the Fe(II) centre. The apical thiocarbonyl ligand displays a linear Fe-C-S linkage (176.3(2)°) with an Fe-C distance of 1.662(3) Å and the mean Fe-N distance is 1.982(5) Å. The low-spin Fe(II) atom is displaced by 0.23 Å from the mean plane of the porphyrinato core.

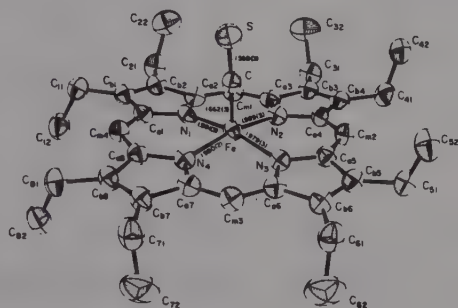


Fig. 1. The  $\text{Fe}(\text{C}_{36}\text{H}_{44}\text{N}_4)(\text{CS})$  molecule.

3,7-DIMETHYL-3',7'-DIHYDRO-2,2',8,8',12,13,17,18-OCTAETHYLPORPHYRIN



K.M. BARKIGIA, J. FAJER, C.K. CHANG and G.J.B. WILLIAMS, 1982. *J. Am. Chem. Soc.*, 104, 315-317.

Triclinic,  $\bar{P}1$ ,  $a = 12.300$ ,  $b = 13.998$ ,  $c = 11.392$  Å,  $\alpha = 109.78$ ,  $\beta = 95.37$ ,  $\gamma = 68.69^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.092$  for 2041 reflexions.

The molecule (Fig. 1) has approximate mirror symmetry across a line through C5 and C15. The reduced rings (containing N(1) and N(2)) are markedly non-planar although the four N atoms are coplanar. Bond distances ( $\sigma = 0.007$ -0.008 Å) are as expected.

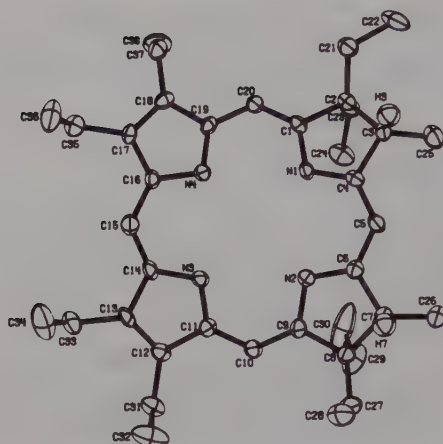


Fig. 1. The  $C_{39}H_{54}N_4$  molecule.

BENZOYL(2,7,12,17-TETRAETHYL-3,8,13,18-TETRAMETHYL-21H,23H-PORPHINATO) RHODIUM(III)  
 $C_{39}H_{41}N_4ORh$

R. GRIGG, J. TROCHA-GRIMSHAW and K. HENRICK, 1982. *Acta Cryst.*, **B38**, 2455-2458.

Triclinic,  $C\bar{1}$ ,  $a = 12.300$ ,  $b = 21.621$ ,  $c = 13.185$  Å,  $\alpha = 90.79$ ,  $\beta = 111.13$ ,  $\gamma = 91.39^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.053$  for 3164 reflexions.

The molecule exists as a discrete five-coordinate approximately square-pyramidal metalloporphyrin with the benzoyl residue as the axial ligand (Fig. 1). Rh lies 0.096 Å out of the plane of the four pyrrole N atoms, away from the benzoyl residue. Interatomic distances are: Rh-C 1.963(7), Rh-N 2.039(6), 2.031(6), 2.031(6) and 2.025(6) Å.

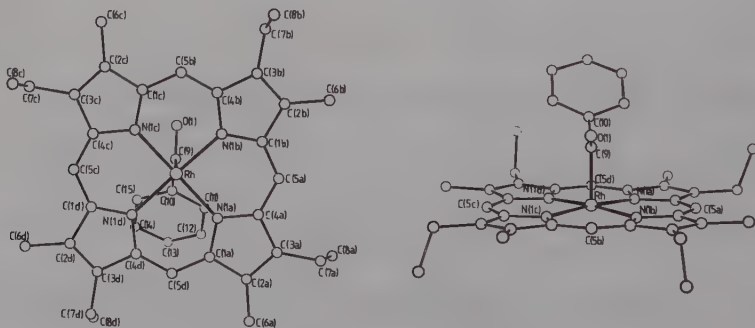


Fig. 1.  $C_{39}H_{41}N_4ORh$ : perspective views of the molecule.

(ISOTHIOCYANATO) (OCTAETHYLPORPHINATO) (PYRIDINE) IRON(III)  
 $C_{42}H_{49}FeN_6S$  (I)

(ISOTHIOCYANATO) (meso-TETRAPHENYLPORPHINATO) (PYRIDINE) IRON(III) PYRIDINE  
 $C_{50}H_{33}FeN_6S \cdot 0.5(C_5H_5N)$  (II)

W.R. SCHEIDT, Y.J. LEE, D.K. GEIGER, K. TAYLOR and K. HATANO, 1982. *J. Am. Chem. Soc.*, **104**, 3367-3374.

I. Triclinic,  $P\bar{1}$ ,  $a = 12.348$ ,  $b = 15.625$ ,  $c = 10.535$  Å,  $\alpha = 92.30$ ,  $\beta = 105.10$ ,  $\gamma = 101.10^\circ$ ,  $D_m = 1.255$ ,  $Z = 2$ . Mo radiation,  $R = 0.052$  for 7549 reflexions.

II. Monoclinic,  $P2_1/n$ ,  $a = 13.238$ ,  $b = 23.917$ ,  $c = 14.269$  Å,  $\beta = 104.74^\circ$ ,  $D_m = 1.294$ ,  $Z = 4$ . Mo radiation,  $R = 0.066$  for 5878 reflexions.

The two molecules are shown in Fig. 1. The high-spin Fe(III) complex I has mean Fe-N<sub>p</sub> 2.048(4), Fe-N(py) 2.442(2) and Fe-N(NCS) 2.031(2) Å with the iron atom displaced 0.24 Å from the mean plane of the core toward the NCS ligand. The Fe-N-C-S group is linear in I. In the low-spin Fe(III) complex II the Fe-N bonds are shorter than those in I by an average of 0.115 Å; mean Fe-N<sub>p</sub> 1.988(9), Fe-N(py) 2.082(3) and Fe-N(NCS) 1.942(4) Å. The Fe-N-C-S group is partly bent, Fe-N-C 155.6(3)°, presumably as a result of packing forces.

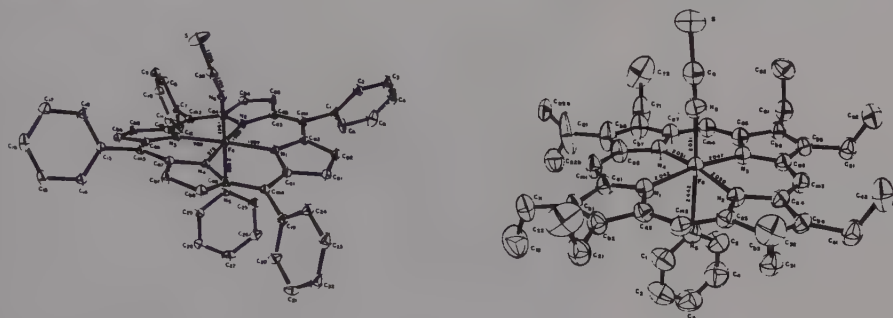


Fig. 1. The  $C_{44}H_{49}FeN_6S$  (left) and  $C_{50}H_{33}FeN_6S$  (right) molecules.

DIAQUA(TETRAPHENYL)PORPHINATO-COBALT(III) PERCHLORATE ACETONE HYDRATE  
 $C_{44}H_{32}ClCoN_4O_6 \cdot 1.5(H_2O) \cdot C_3H_6O$  [Co(tpp)(H<sub>2</sub>O)<sub>2</sub>], ClO<sub>4</sub>, C<sub>3</sub>H<sub>6</sub>O, 1.5(H<sub>2</sub>O)

H. MASUDA, T. TAGA, K. OSAKI, H. SUGOMOTO and M. MORI, 1982. Bull. Chem. Soc. Jpn., 55, 4-8.

Monoclinic,  $P2_1/c$ ,  $a = 13.93$ ,  $b = 23.46$ ,  $c = 17.35$  Å,  $\beta = 129.4^\circ$ ,  $D_m = 1.33$ ,  $Z = 4$ . Mo radiation,  $R = 0.077$  for 4380 reflexions.

The Co atom lies in the mean plane of the porphyrinato core and has octahedral coordination (Fig. 1) with mean Co-N 1.964(4), Co-O(H<sub>2</sub>O) 1.936(5) Å. The porphyrin plane is ruffled and has approximate 4 symmetry. The ions and solvate molecules are linked by hydrogen bonds.

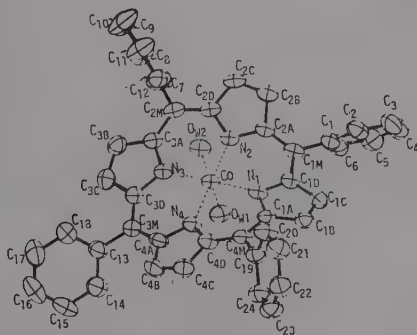


Fig. 1. A view of  $[Co(tpp)(H_2O)_2]^+$ .

(OCTAETHYLPORPHINATO)BIS(TETRAHYDROFURAN)IRON(III) PERCHLORATE  
 $C_{44}H_{60}ClFeN_4O_6$



H. MASUDA, T. TAGA, K. OSAKI, H. SUGIMOTO, Z.-I. YOSHIDA and H. OGOSHI, 1982.  
 Bull. Chem. Soc. Jpn., 55, 3891-3895.

Monoclinic,  $C2/m$ ,  $a = 13.916$ ,  $b = 16.509$ ,  $c = 10.659$  Å,  $\beta = 118.18^\circ$ ,  $D_m = 1.24$ ,  $Z = 2$ . Mo radiation,  $R = 0.079$  for 597 reflexions.

The molecule (Fig. 1) has crystallographic  $2/m$  symmetry (the mirror plane passes through Fe, C(5) and O(T)). The porphinato core is planar to within 0.06 Å. The Fe atom has octahedral coordination with Fe-O 2.187(11) and Fe-N 1.978(12) Å. These dimensions are explained in terms of an intermediate-spin state of the iron(III) ion.

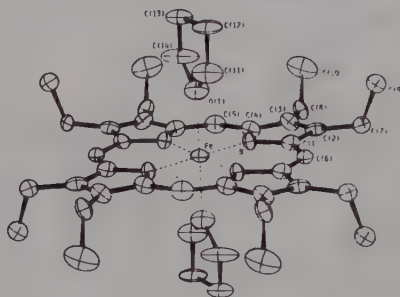


Fig. 1. A view of  $[Fe(oep)(thf)_2]^+$ .

N-METHYL-5,10,15,20-TETRAKIS(p-BROMOPHENYL)PORPHYRIN DICHLOROMETHANE SOLVATE  
 $C_{45}H_{28}Br_4N_4, CH_2Cl_2$

D.K. LAVALLEE and O.P. ANDERSON, 1982. J. Am. Chem. Soc., 104, 4707-4708.

Monoclinic,  $P2_1/c$ ,  $a = 15.440$ ,  $b = 16.261$ ,  $c = 17.534$  Å,  $\beta = 108.16^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.082$  for 3860 reflexions.

The molecule (Fig. 1) displays distortions from planarity of the porphyrin core as a result of N-methylation. The substituted pyrrole ring is canted  $27.7^\circ$  from the N1-N2-N3 plane, adjacent rings being canted by  $10.2$  and  $11.9^\circ$  in the opposite direction. The opposite ring is canted  $8.1^\circ$  in the same direction as the substituted ring. No unusual bond lengths were noted.

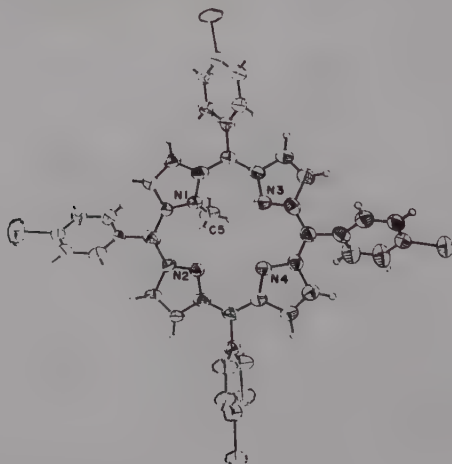


Fig. 1. The  $C_{45}H_{28}Br_4N_4$  molecule.

DIMETHOXY(5,10,15,20-TETRAPHENYLPORPHINATO)MANGANESE(IV) METHANOL SOLVATE

$C_{46}H_{34}MnN_4O_2 \cdot 0.25(CH_3O)$

$Mn(C_{44}H_{28}N_4)(OCH_3)_2 \cdot 0.25(CH_3OH)$

M.J. CAMENZIND, F.J. HOLLANDER and C.L. HILL, 1982. *Inorg. Chem.*, **21**, 4301-4308.

Tetragonal,  $P4_2/n$ ,  $a = 19.388$ ,  $c = 9.7794$  Å,  $Z = 4$ . Mo radiation,  $R = 0.042$  for 1395 reflexions.

The molecule is shown in Fig. 1. The Mn atom is located at a crystallographic inversion centre and is six-coordinate with the O-Mn-O unit linear and the  $MnN_4$  unit strictly planar. Bond lengths: Mn-O 1.839(2), Mn-N1 1.993(3), Mn-N2 2.031(3), O-C 1.387(3) Å.

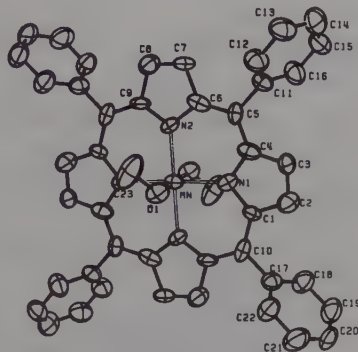


Fig. 1. The  $Mn(C_{44}H_{28}N_4)(OCH_3)_2$  molecule.

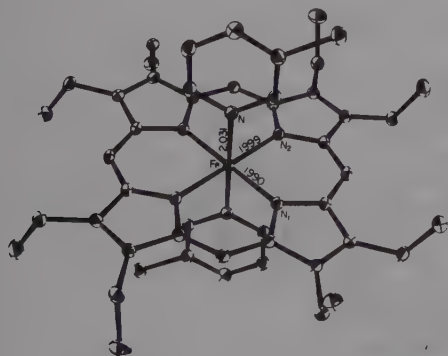
BIS(3-CHLOROPYRIDINE) (OCTAETHYLPORPHINATO) IRON(III) PERCHLORATE

$C_{46}H_{52}Cl_3FeN_6O_4$

$[Fe(C_{36}H_{44}N_4)(3-ClC_5H_4N)_2](ClO_4)$

W.R. SCHEIDT, D.K. GEIGER and K.J. HALLER, 1982. *J. Am. Chem. Soc.*, **104**, 495-499.

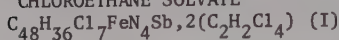
Triclinic,  $P\bar{1}$ ,  $a = 10.798$ ,  $b = 11.400$ ,  $c = 9.740$  Å,  $\alpha = 113.08$ ,  $\beta = 95.66$ ,  $\gamma = 72.87^\circ$ ,  $Z = 1$ . Mo radiation,  $R = 0.043$  for 5315 reflexions at 98 K.  $a = 10.929$ ,  $b = 11.480$ ,  $c = 9.954$  Å,  $\alpha = 112.27$ ,  $\beta = 94.30$ ,  $\gamma = 73.34^\circ$ ,  $D_m = 1.39$ ,  $Z = 1$ . Mo radiation,  $R = 0.045$  for 5228 reflexions at 293 K.



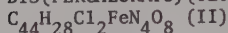


in the low-spin state, the average Fe-N(porph) distance is 1.994(6) Å and the axial Fe-N distance is 2.031(2) Å. At 293 K, where there is a 55:45 thermal mixture of high- and low-spin states, the "average" Fe-N(p) and Fe-N(axial) distances are 2.014 and 2.194(2) Å (consistent with 55% high-spin state). A crystallographic resolution of spin isomers at 293 K was accomplished. This gives resolved (3-ClC<sub>5</sub>H<sub>4</sub>N) ligand positions which lead to low-spin and high-spin axial Fe-N distances of 2.043 and 2.316 Å. No motion of the Fe(III) ion is required for the spin-state transition.

CHLORO(TETRA-*p*-TOLYLPORPHYRINATO)IRON(III) HEXACHLOROANTIMONY(V) 1,1,2,2-TETRA-CHLOROETHANE SOLVATE



BIS(PERCHLORATO)(TETRAPHENYLPORPHYRINATO)IRON(III)



G. BUISSON, A. DERONZIER, E. DUÉE, P. GANS, J.-C. MARCHON and J.-R. REGNARD, 1982. J. Am. Chem. Soc., 104, 6793-6796.

I. Monoclinic,  $P2_1/c$ ,  $a = 10.98$ ,  $b = 22.57$ ,  $c = 23.65$  Å,  $\beta = 97.73^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.088$  for 4627 reflexions (at  $-140^\circ\text{C}$ ).

II. Monoclinic,  $P2_1/c$ ,  $a = 12.132$ ,  $b = 14.622$ ,  $c = 13.153$  Å,  $\beta = 127.84^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.116$  for 1448 reflexions.

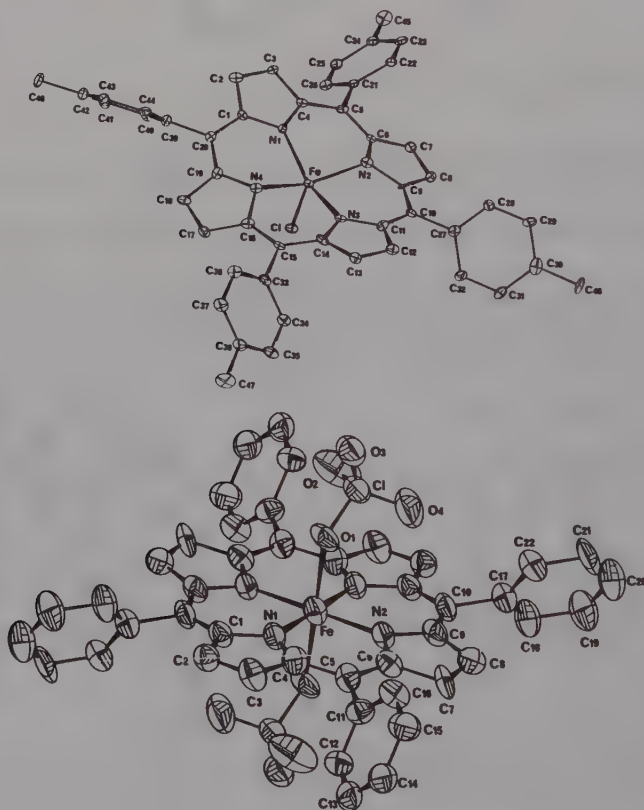


Fig. 1. The  $[(\text{C}_{48}\text{H}_{36}\text{N}_4)\text{FeCl}]^+$  cation (top) and the  $(\text{C}_{44}\text{H}_{28}\text{N}_4)\text{Fe}(\text{ClO}_4)_2$  molecule (bottom).

The two Fe(III) porphyrin  $\pi$ -cation radical complexes are shown in Fig. 1. The most striking feature of the cation in I is the conformation of the porphyrin, in which nearly planar pyrrole rings tilt alternately up and down to give rise to a saddle-shaped macrocycle. Bond lengths in I (Fe-N 2.05(1)-2.09(1), Fe-Cl 2.168(5) Å) are consistent with a high-spin Fe(III) formulation. In the centrosymmetric octahedral complex II the Fe atom is coordinated by four N and two perchlorate O atoms. The macrocycle is planar, and the distances Fe-N 2.04(1) and 2.05(1), Fe-O 2.13(1) Å again confirm the Fe(III) state.

NITRIDO[TETRAKIS(p-METHOXYPHENYL)PORPHINATO]MANGANESE(V) CHLOROBENZENE SOLVATE  
 $C_{48}H_{36}MnN_5O_4 \cdot x(C_6H_5Cl)$

C.L. HILL and F.J. HOLLANDER, 1982. J. Am. Chem. Soc., 104, 7318-7319.

Monoclinic, C2/c, a = 30.5128, b = 9.5508, c = 15.2593 Å,  $\beta = 92.7^\circ$ , Z = 4.  
 Mo radiation, R = 0.042 for 2117 reflexions.

The molecule (Fig. 1) occupies a centre of symmetry, the Mn $\equiv$ N moiety being disordered 50-50 above and below the  $N_h$  plane. The porphyrin core is planar to within 0.06 Å and Mn is 0.388 Å out of the  $N_h$  plane. Bond lengths: Mn-N 1.983(2)-2.060(2), Mn $\equiv$ N 1.515(3) Å.

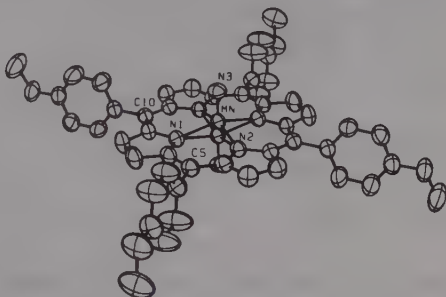


Fig. 1. The  $C_{48}H_{36}MnN_5O_4$  molecule.

OXO(5,10,15,20-TETRA-p-TOLYLPORPHYRINATO)CHROMIUM(IV)  
 $C_{48}H_{38}CrN_4O$

J.T. GROVES, W.J. KRUPER, Jr., R.C. HAUSHALTER and W.M. BUTLER, 1982. Inorg. Chem., 21, 1363-1368.

Monoclinic, P2<sub>1</sub>/c, a = 17.342, b = 16.964, c = 15.804 Å,  $\beta = 112.52^\circ$ , Z = 4. Mo radiation, R = 0.068 for 2309 reflexions.

The molecule (Fig. 1) contains a square-pyramidal Cr(IV) ion with an apical Cr-O bond of 1.572(6) Å, mean Cr-N 2.032(7) Å. The Cr atom is displaced 0.469 Å above the basal plane and the porphyrin ring is distinctly saddle-shaped with  $\beta$ -carbons displaced 0.340 and 0.568 Å above and below the  $N_h$  mean plane.

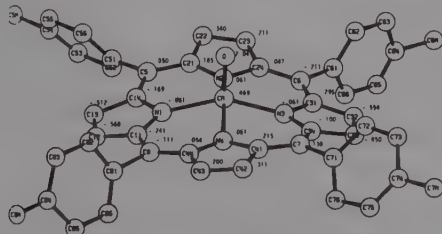


Fig. 1. The  $C_{48}H_{38}CrN_4O$  molecule.

(N,N'-(BENZYLOXY)METHYLENE)TETRAPHENYLPORPHYRIN-N'',N''')DIBROMOPALLADIUM(II)  
DICHLOROMETHANE

$C_{52}H_{36}Br_2N_4OPd, CH_2Cl_2$

$PdBr_2(N_4OC_{52}H_{36}), CH_2Cl_2$

H.J. CALLOT, J. FISCHER and R. WEISS, 1982. J. Am. Chem. Soc., 104, 1272-1276.

Triclinic,  $P1$ ,  $a = 10.591$ ,  $b = 10.706$ ,  $c = 12.560$  Å,  $\alpha = 118.82$ ,  $\beta = 114.63$ ,  $\gamma = 77.68^\circ$ ,  $D_m = 1.56$ ,  $Z = 1$ . Cu radiation,  $R = 0.051$  for 3569 reflexions.

The (benzyloxy)methylene moiety bridges two adjacent N atoms and the  $PdBr_2$  group bridges the two other N atoms leading to a very distorted porphyrin ring. The Pd atom is out of the 4N plane by 1.460 Å, and the individual pyrrole rings make dihedral angles with the 4N plane between  $8.2$  and  $21.3^\circ$ . Pd-Br 2.395(1), 2.429(1); Pd-N 2.010(7), 2.071(7) Å.

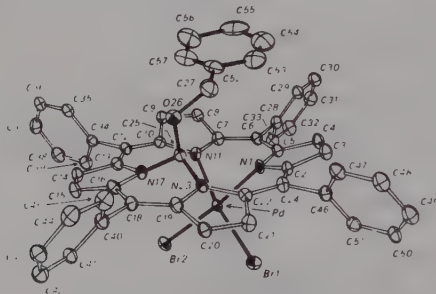


Fig. 1. The  $PdBr_2(N_4OC_{52}H_{36})$  molecule.

HEPTAMETHYL DICYANOCOBYRINATE (COBESTER)

$C_{54}H_{73}CoN_6O_{14}$

K. KAMIYA and O. KENNARD, 1982. J. Chem. Soc. Perkin I, 2279-2288.

Orthorhombic,  $P2_12_12_1$ ,  $a = 18.931$ ,  $b = 18.960$ ,  $c = 15.643$  Å,  $Z = 4$ . Mo radiation,  $R = 0.132$  for 2253 reflexions.

The side chains at C(3), C(8), C(13) and C(17) (Fig. 1) are all disordered to varying degrees. The Co atom is co-ordinated in a distorted octahedron with approximate local symmetry  $mm2$ . The twofold axis passes through C(10) and Co. Bond lengths to the Co atom and the N-Co-N bond angles are not significantly different from those in a number of related compounds, including the hydrated form of cobester (1). N(1)-Co-N(4), the angle opposite the direct pyrrole junction, is significantly less than a right angle at  $81.0^\circ$  while N(2)-Co-N(3) is larger ( $97.2^\circ$ ). N(1)-Co-N(2) ( $91.8$ ) and N(3)-Co-N(4) ( $90.4^\circ$ ) are equal within experimental error. The co-ordination around the Co atom is markedly nonplanar with N(2) and N(4) above the mean plane and N(1) and N(3) below it. There are no significant short contacts in the crystal.

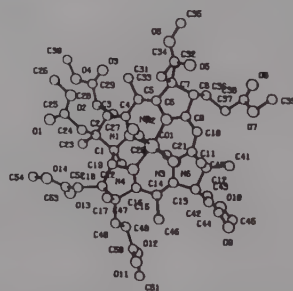
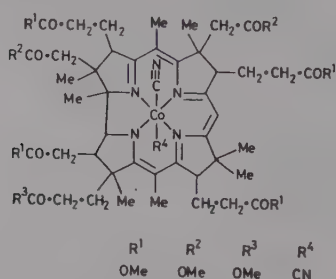


Fig. 1. Molecular skeleton and structure of  $C_{54}H_{73}CoN_6O_{14}$ .

# 1. Structure Reports, 46B, 508.

[5,10,15,20-[PYRROMELLITOYL TETRAKIS(o-(OXYETHOXY)PHENYL)]PORPHYRINATO]CHLOROIRON(III)  
 CHLOROFORM  
 $C_{62}H_{42}ClFeN_4O_{12} \cdot 3(CHCl_3)$

M. SABAT and J.A. IBERS, 1982. J. Am. Chem. Soc., 104, 3715-3721.

Monoclinic,  $P2_1/c$ ,  $a = 14.473$ ,  $b = 19.514$ ,  $c = 22.830$  Å,  $\beta = 101.61^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.065$  for 9897 reflexions (at  $-150^\circ C$ ).

The "capped" porphyrin is shown in Fig. 1. The  $Cl^-$  ligand is outside the "cap" and the iron atom is above the mean plane of the porphyrin core away from the "cap". The geometry about the Fe atom is distorted square pyramidal with Fe-N 2.059(3)-2.065(3), Fe-Cl 2.241(1) Å, and Fe displaced 0.46 Å above the mean  $N_4$  plane.

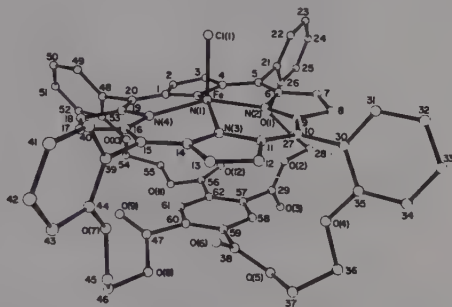


Fig. 1. The  $C_{62}H_{42}ClFeN_4O_{12}$  molecule.

CHLORO-(2,2-BIS(p-CHLOROPHENYL VINYLIDENE-N,Fe)-5,10,15,20-TETRA-p-TOLYL PORPHINATO)IRON  
 DICHLOROMETHANE SOLVATE  
 $C_{62}H_{44}Cl_3FeN_4 \cdot 2(CH_2Cl_2)$   $(C_{48}H_{36}N_4)[(ClC_6H_4)_2C=C]FeCl, 2CH_2Cl_2$

M.M. OLMSTEAD, R.-J. CHENG and A.L. BALCH, 1982. Inorg. Chem., 21, 4143-4148.

Monoclinic,  $P2_1/n$ ,  $a = 19.653$ ,  $b = 12.418$ ,  $c = 23.473$  Å,  $\beta = 103.10^\circ$ ,  $D_m = 1.35$ ,  $Z = 4$ . Mo radiation,  $R = 0.061$  for 6106 reflexions (at 140 K,  $D_m$  at 298 K).

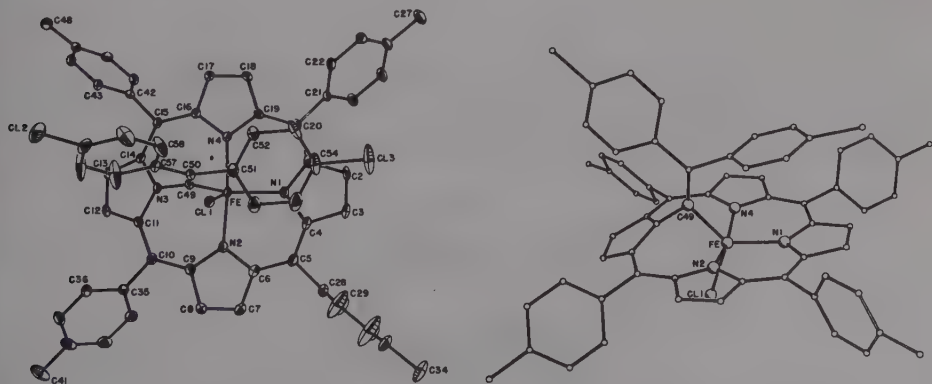


Fig. 1. Two views of the  $(C_{48}H_{36}N_4)[(ClC_6H_4)_2C=C]FeCl$  molecule.

The molecule, in which the vinylidene carbene ligand has inserted into an iron-nitrogen bond, is shown in Fig. 1. The Fe atom is five-coordinate with approximate trigonal bipyramidal geometry. Bond lengths: Fe-N (axial) 1.991(4) and 1.985(4), Fe-N (equatorial) 2.002(4), Fe-C 1.921(5) and Fe-Cl 2.299(1) Å.

$\mu_2$ -OXO-BIS[(OCTAETHYLPORPHINATO)-CHLORO-RUTHENIUM(IV)] BENZENE SOLVATE  
 $C_{72}H_{88}Cl_2N_8ORu_2 \cdot C_6H_6$  [Ru(oep)Cl] $_2$ O,  $C_6H_6$

H. MASUDA, T. TAGA, K. OSAKI, H. SUGIMOTO, M. MORI and H. OGOSHI, 1982. Bull. Chem. Soc. Jpn., 55, 3887-3890.

Tetragonal, P4/nnc,  $a = 13.912$ ,  $c = 17.821$  Å,  $D_m = 1.300$ ,  $Z = 2$ . Mo radiation,  $R = 0.084$  for 958 reflexions.

The molecule (Fig. 1) has crystallographic 422 ( $D_4$ ) symmetry and contains the binuclear (Cl)N<sub>4</sub>Ru-O-RuN<sub>4</sub>(Cl) coordination group with Ru-O( $\mu$ ) 1.793(2) Å, Ru-Cl 2.320(6) Å, and Ru-N 2.038(9) Å. The Ru-O-Ru bond is linear, and the two porphinato cores rotate around the Ru-O-Ru bond by 21.0° from the eclipsed form. The porphinato core is planar within 0.04 Å, and the Ru atom is displaced only by 0.07 Å out of the plane defined by the four porphinato nitrogen atoms toward the bridging O atom.

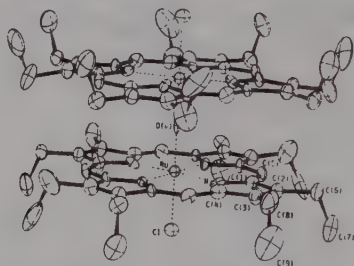


Fig. 1. A view of the [Ru(oep)Cl] $_2$ O molecule.

$\mu$ -OXO-BIS[AZIDO(TETRAPHENYLPORPHINATO)MANGANESE(IV)] CHLOROBENZENE SOLVATE  
 $C_{88}H_{56}Mn_2N_{14}O \cdot 1.5(C_6H_5Cl)$

B.C. SCHARDT, F.J. HOLLANDER and C.L. HILL, 1982. J. Am. Chem. Soc., 104, 3964-3972.

Orthorhombic, Pbcn,  $a = 21.208$ ,  $b = 16.826$ ,  $c = 22.620$  Å,  $D_m = 1.36$ ,  $Z = 4$ . Mo radiation,  $R = 0.094$  for 3276 reflexions.

The molecule (Fig. 1) possesses exact  $C_2$  symmetry with both Mn atoms, the  $\mu$ -O atom, and the ligating azido N atoms lying on the twofold axis. The two Mn atoms are displaced from the mean N<sub>4</sub> planes toward the bridging O atom by 0.10 and 0.08 Å. The mean Mn-N(porphyrin) distance is 2.014(19), Mn-O 1.743(4) and 1.794(7), Mn-N(axido) 1.998(8) and 1.993(7) Å, Mn-O-Mn 180°, Mn-N-N 128.3(7) and 125.0(5)°.

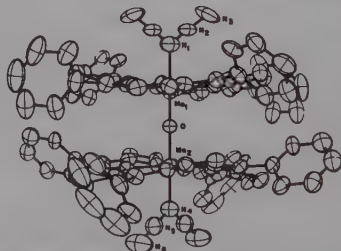


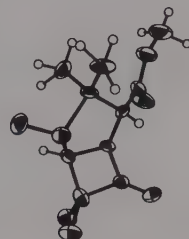
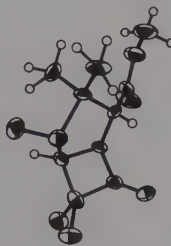
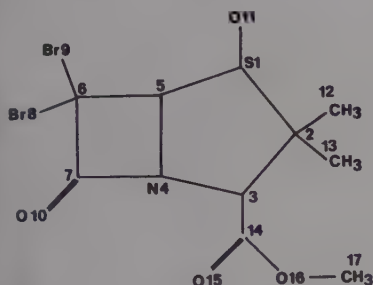
Fig. 1. The  $C_{88}H_{56}Mn_2N_{14}O$  molecule ( $N_3$  ligands are twofold disordered).



## 6,6-DIBROMOMETHYLPENICILLANATE-4-OXIDE

 $C_9H_{11}Br_2NO_4S$ O.M. PEETERS, N.M. BLATON, C.J. DE RANTER, 1982. Cryst. Struct. Comm., 11, 787-791.Orthorhombic,  $P2_12_12_1$ ,  $a = 6.232$ ,  $b = 9.420$ ,  $c = 22.815$  Å,  $D_m = 1.91$ ,  $Z = 4$ . Cu radiation,  $R = 0.070$  for 999 reflexions.

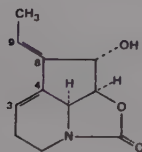
The thiazolidine ring (Fig. 1) has an envelope conformation with the sulphur atom 0.94 Å out of the plane of the remaining four atoms. The  $\beta$ -lactam nitrogen atom is 0.35 Å out of the plane of its substituents and C(5) is 0.26 Å from the N4,C6,C7,O10 plane. The sulphur has configuration R.

Fig. 1. The  $C_9H_{11}Br_2NO_4S$  molecule and a stereoview.

## O-ACETYLDIHYDROSTREPTAZOLIN

 $C_{13}H_{17}NO_4$ A. KARRER and M. DOBLER, 1982. Helv. Chim. Acta, 65, 1432-1435.Orthorhombic,  $P2_12_12_1$ ,  $a = 8.628$ ,  $b = 9.814$ ,  $c = 14.932$  Å,  $Z = 4$ . Mo radiation,  $R = 0.047$  for 1240 reflexions.

The analysis established the constitution and stereochemistry as shown in Fig. 1. The N atom is pyramidal and lies 0.29 Å above the C(1),C(5),C(11) plane.





Tetragonal,  $P4_1$ ,  $a = 8.967$ ,  $c = 19.904 \text{ \AA}$ ,  $D_m = 1.40$ ,  $Z = 4$ . Cu radiation,  $R = 0.056$  for 1840 reflexions.

The molecular conformation (Fig. 1) is significantly different from that of the 5-bromo derivative (1). The methoxy substituents on the benzene ring are coplanar with the ring rather than clinal to it as in the 5-bromo derivative. The five-membered ring is only slightly puckered and the cyclohexanone ring has a half-chair conformation with the 6'-methyl group oriented over the five-membered ring to a greater extent in griseofulvin than in the 5-bromo derivative.

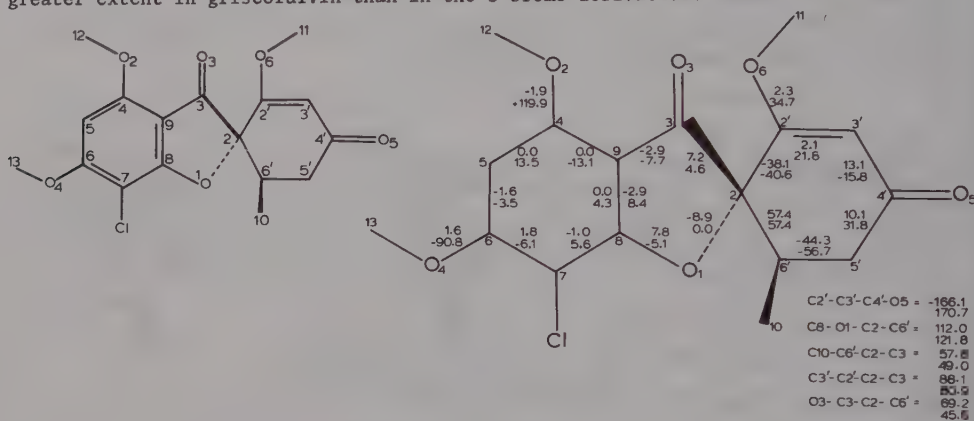


Fig. 1. Griseofulvin and torsion angles (lower values are for 5-bromo-griseofulvin).

1. Structure Reports, 28, 533.

2,2,2-TRICHLOROETHYL 6 $\alpha$ -ALLYL-6 $\beta$ -PHENYLTHIOPENICILLANATE  
 $C_{19}H_{20}Cl_3NO_3S_2$

P.J. GIDDINGS, D.I. JOHN, E.J. THOMAS and D.J. WILLIAMS, 1982. J. Chem. Soc. Perkin I, 2757-2766.

Orthorhombic,  $P2_12_12$ ,  $a = 19.710$ ,  $b = 17.486$ ,  $c = 6.496 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.040$  for 1686 reflexions. [The crystals deteriorated slowly during data collection.]

The X-ray structure was used to assign the absolute configuration at C(6) (Fig. 1). The bond lengths in the  $\beta$ -lactam ring are N(4)-C(5) 1.463, C(5)-C(6) 1.555, C(6)-C(7) 1.528, C(7)-N(4) 1.386 and the C(7)-O(7) carbonyl bond is 1.198  $\text{\AA}$ . The angles in the ring are N(4)-C(5)-C(6) 88.2, C(5)-C(6)-C(7) 85.2, C(6)-C(7)-N(4) 92.1 and C(7)-N(4)-C(5) 94.2 $^\circ$ .

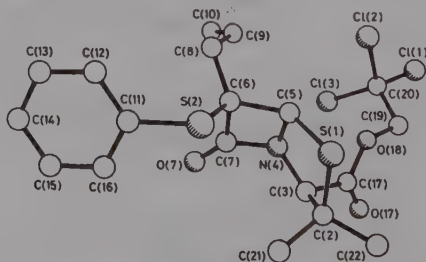


Fig. 1. Molecular structure and absolute configuration of  $C_{19}H_{20}Cl_3NO_3S_2$ .

(6R,7R)-7-[(R)-2-CARBOXY-2-(4-HYDROXYPHENYL)ACETAMIDO]-7-METHOXY-3-[[[1-METHYL-1H-TETRAZOL-5-YL)THIO]METHYL]-8-OXO-5-OXA-1-AZABICYCLO[4.2.0]OCT-2-ENE-2-CARBOXYLIC ACID DIAMMONIUM SALT HYDRATE  
 $C_{20}H_{26}N_8O_9S \cdot 4.5(H_2O)$

M. SHIRO, H. NAKAI, F. MATSUBARA and I. KIKKAWA, 1982. Cryst. Struct. Comm., **11**, 727-732.

Triclinic,  $P1$ ,  $a = 10.580$ ,  $b = 14.820$ ,  $c = 9.703$  Å,  $\alpha = 94.00$ ,  $\beta = 102.90$ ,  $\gamma = 84.80^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.031$  for 5121 reflexions.

The two independent anions have almost the same conformation; one is shown in Fig. 1. N(5) of molecule A is 0.184(2) Å out of the plane of C4,C6,C8 (0.168 Å in molecule B). Bond length data are consistent with the non-planarity of the  $\beta$ -lactam N atom reducing amide resonance.

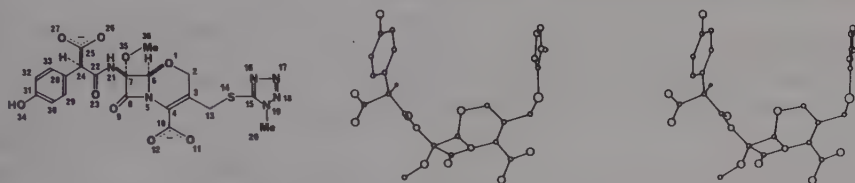


Fig. 1. The  $C_{20}H_{18}N_6O_9^{2-}$  ion, and a stereoview of molecule A.

#### MYCINOLIDE IV

$C_{21}H_{32}O_5$

M. HAYASHI, K. KINOSHITA, S. SATOI and K. NAKATSU, 1982. J. Antibiotic, **35**, 1243-1244.

Orthorhombic,  $P2_12_12$ ,  $a = 11.533$ ,  $b = 30.651$ ,  $c = 6.101$  Å,  $Z = 4$ . Mo radiation,  $R = 0.095$  for 1074 reflexions.

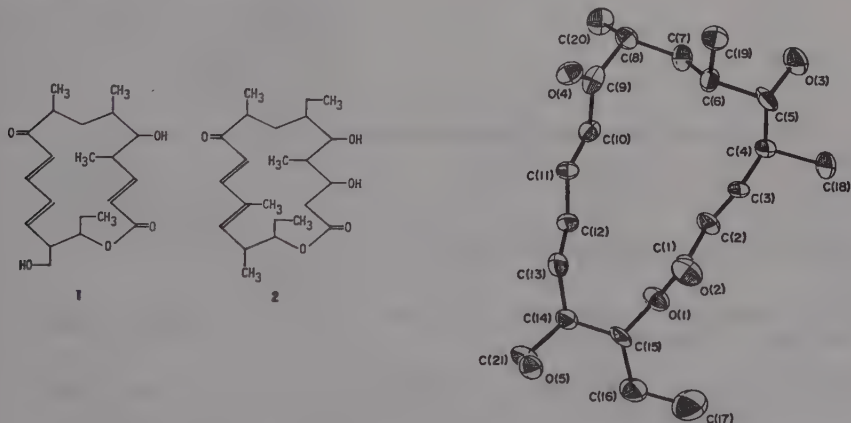


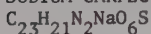
Fig. 1. Schematic figures of  $C_{21}H_{32}O_5$  (1) and protylonolide (2) (left), and molecular structure of  $C_{21}H_{32}O_5$  (right).

The conformation of the 16-membered macrolide, mycinolide IV (Fig. 1) is very similar to that of the compound protylonolide (1). Aside from dissimilarities arising from the structural difference between  $C(2)=C(3)$  in the former and  $C(2)-C(3)(OH)-$  in the latter, the torsion angles are very similar and hence the conformations of the macro-rings. Selected torsion angles are:  $C(4)-C(5)-C(6)-C(7)$  -69

and  $-72^\circ$ , C(8)-C(9)-C(10)-C(11)  $+169$  and  $+168^\circ$ , C(12)-C(13)-C(14)-C(15)  $+94$  and  $+98^\circ$ , C(6)-C(7)-C(8)-C(9)  $-56$  and  $-61^\circ$ , O(1)-C(1)-C(2)-C(3)  $-177$  and  $+150^\circ$ , and C(2)-C(3)-C(4)-C(5)  $+144$  and  $+179^\circ$  for mycinolide IV and protylonolide respectively.

1. Structure Reports, 46B, 517.

SODIUM CARFECILLIN



P.A.C. GANE, M.O. BOLES and A.E. BIRD, 1982. Acta Cryst., B38, 929-932.

Monoclinic,  $P2_1$ ,  $a = 8.77$ ,  $b = 6.20$ ,  $c = 21.40 \text{ \AA}$ ,  $\beta = 99.5^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.095$  for 1198 reflexions (photographic data).

In the molecule the S-C bond lengths (1.907 and 1.816  $\text{\AA}$ ) differ significantly but most other bond lengths are as expected. The configuration of the asymmetric carbon to which the phenyl ester group is attached is R. The crystal structure (Fig. 1) is compared with that of other penicillins.

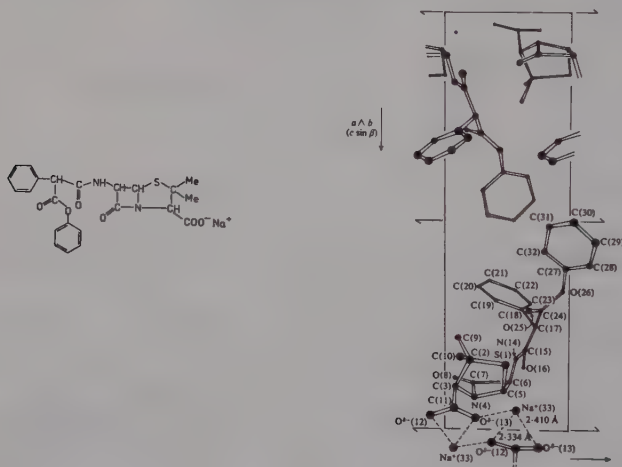


Fig. 1. The crystal structure of sodium carfecillin viewed along  $a$ .

ACETYLASPARENOMYCIN A p-NITROBENZYLACETATE



N. TSUJI, K. NAGASHIMA, M. KOBAYASHI, J. I. SHOJI, T. KATO, Y. TERUI, N. NAKAI and M. SHIRO, 1982. J. Antibiotics, 35, 24-31.

Orthorhombic,  $P2_12_12_1$ ,  $a = 19.249$ ,  $b = 25.361$ ,  $c = 4.952 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.036$  for 2372 reflexions.

The absolute configuration of the title compound is shown in Fig. 1. It indicates that the S(9) atom adopts the R configuration and that the bond C(6)=C(8) exhibits E geometry. The N(1) atom deviates from the plane of C(2), C(5) and C(7) by 0.538  $\text{\AA}$ . The bond lengths in the  $\beta$ -lactam amide group (N(1)-C(7) 1.437; C(7)-O(23) 1.196  $\text{\AA}$ ) differ from those in the exocyclic amide group (N(12)-C(13) 1.370; C(13)-O(14) 1.228  $\text{\AA}$ ).

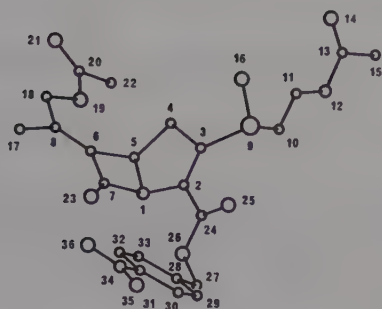


Fig. 1. Perspective view of the molecule of  $C_{23}H_{23}N_3O_9S$ .

4-METHOXY-5-METHYL-6-(7,9,11-TRIMETHYL-1,3,5,7,9,11-TRIDECAHEXAENYL)-2H-PYRAN-2-ONE  
(CITREOMONTANINE)

$C_{23}H_{28}O_3$

C. BRASSY, B. BACHET, C. GUIDI-MOROSINI, S. REBUFFAT and D. MOLHO, 1982. *Acta Cryst.*, B38, 1624-1626.

Triclinic,  $P\bar{1}$ ,  $a = 12.092$ ,  $b = 11.018$ ,  $c = 8.018$  Å,  $\alpha = 106.5$ ,  $\beta = 94.8$ ,  $\gamma = 97.7^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.059$  for 2913 reflexions.

In the polyene chain, essentially planar except for atoms C(18), C(19) and C(20), (Fig. 1) the double bonds (mean 1.345(6) Å) have the E configuration and the single bonds (mean 1.444(6) Å) are s-trans except for C(18)-C(19) which is s-cis.

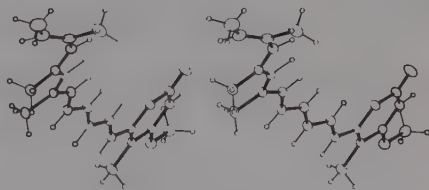
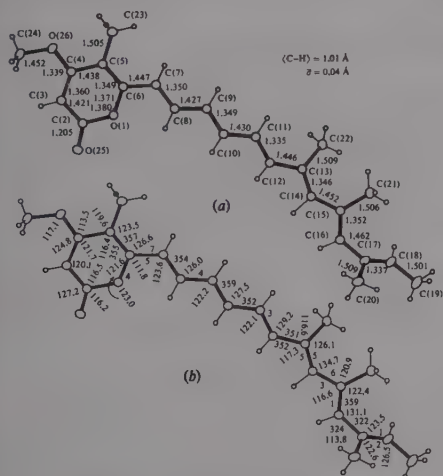


Fig. 1. Bond lengths and angles and torsion angles in citreomontanine, and a stereoview of the molecule.

3',6'-O-(1,1,3,3-TETRAISOPROPYL-1,3-DISILOXANEDIYL)NEPLANOCIN A  
 $C_{23}H_{39}N_5O_4Si_2$

M. YAMAZAKI, K. HAMADA, Y. YAMAGATA, T. FUJIIWARA, K.-I. TOMITA, K. FUKUKAWA, T. UEDA and T. HIRANO, 1982. *Acta Cryst.*, B38, 1176-1180.

Monoclinic,  $P2_1$ ,  $a = 8.953$ ,  $b = 34.638$ ,  $c = 8.957$  Å,  $\beta = 94.41^\circ$ ,  $D_m = 1.215$ ,  $Z = 4$ . Cu radiation,  $R = 0.055$  for 5046 reflexions.

The conformations of the two independent molecules in the asymmetric unit are quite similar, and no significant differences are found in bond distances and angles (Fig. 1). Both the sugar C(2') atoms are displaced by 0.3 Å toward the base N(9), i.e. C(2')-endo puckering, and the torsion angles  $\chi_{CN}$  are  $34.4$  and  $33.0^\circ$  respectively for the two independent molecules (anti conformation).

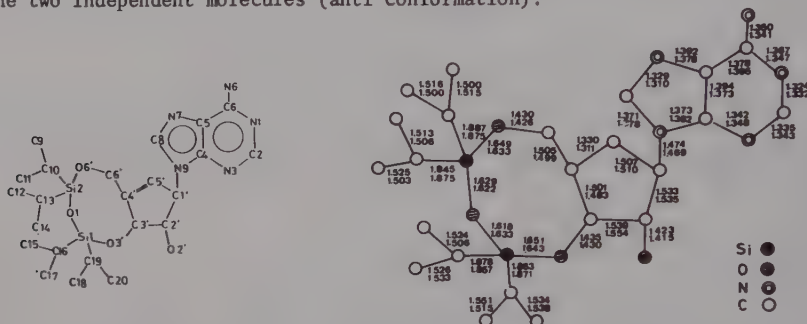


Fig. 1.  $C_{23}H_{39}N_5O_4Si_2$ : bond distances for the two independent molecules (upper and lower values).

17,18-DI-O-ACETYLASPOCHALASIN C BENZENE SOLVATE  
 $C_{28}H_{39}NO_6 \cdot C_6H_6$

K. NEUPERT-LAVES and M. DOBLER, 1982. *Helv. Chim. Acta*, 65, 1426-1431.

Monoclinic,  $C2$ ,  $a = 26.377$ ,  $b = 10.840$ ,  $c = 11.937$  Å,  $\beta = 102.08^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.134$  for 1367 reflexions.

The analysis confirms the chemical constitution and establishes the relative stereochemistry as shown in Fig. 1.

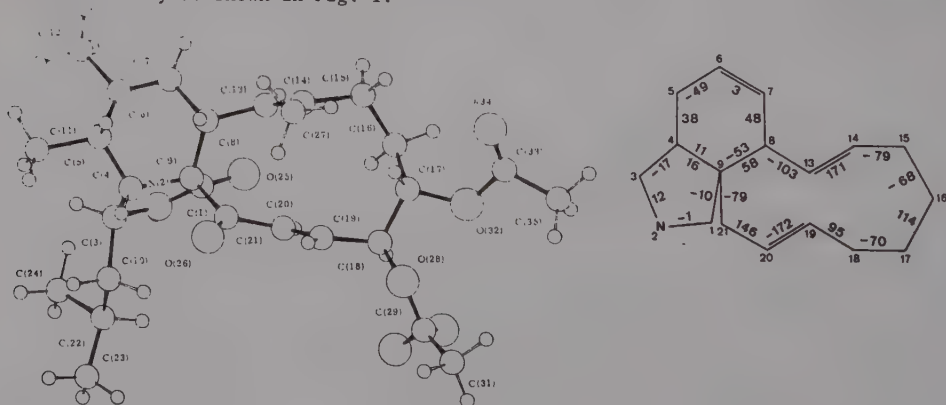


Fig. 1. The 17,18-di-O-acetylaspochalasin C molecule and ring torsion angles.

## PIKROMYCIN

 $C_{28}H_{47}NO_8$ 

A. FURUSAKI, T. MATSUMOTO, K. FURUHATA and H. OGURA, 1982. Bull. Chem. Soc. Jpn., 55, 59-62.

Orthorhombic,  $P2_12_12_1$ ,  $a = 11.295$ ,  $b = 25.734$ ,  $c = 10.509$  Å,  $Z = 4$ . Cu radiation,  $R = 0.056$  for 2259 reflexions.

The conformation of the 14-membered macrocycle is shown in Fig. 1; the C=C and C=O conjugated double bonds are cisoid in the solid state. Molecules are linked by O-H...N (2.829(4) Å) and O-H...O (2.958(4) Å) hydrogen bonds.

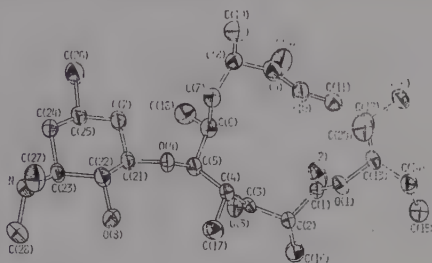


Fig. 1. A view of pikromycin.

## RORIDIN A

 $C_{29}H_{40}O_9$ 

B.B. JARVIS, J.O. MIDIWO, J.L. FLIPPEN-ANDERSON and E.P. MAZZOLA, 1982. J. Nat. Prod., 45, 440-447.

Monoclinic,  $P2_1$ ,  $a = 10.197$ ,  $b = 14.079$ ,  $c = 9.606$  Å,  $\beta = 94.6^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.0487$  for 1890 reflexions.

The analysis establishes the configurations at C6' and C13' in roridin A to be as shown in Fig. 1. The C5-C6 bond (between fully substituted carbon atoms) is somewhat long at 1.579(6) Å, but is not unexpected. The cyclohexene ring is a somewhat flattened half-chair, the central six-membered ring has a slightly distorted chair form, and the five-membered ring is a C(12) envelope.

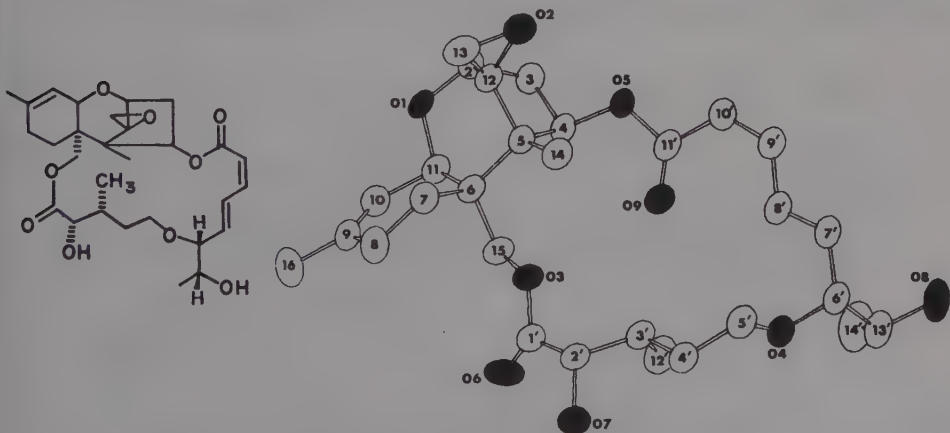
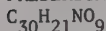


Fig. 1. The roridin A molecule.



## FREDERICAMYCIN A



R. MISRA, R.C. PANDEY and J.V. SILVERTON, 1982. *J. Am. Chem. Soc.*, **104**, 4478-4479.

Orthorhombic,  $P2_12_12_1$ ,  $a = 10.226$ ,  $b = 20.202$ ,  $c = 23.736 \text{ \AA}$ ,  $Z = 8$ . Cu radiation,  $R = 0.052$  for 2407 reflexions.

The two independent molecules are shown in Fig. 1. In each case the two aromatic portions are nearly at right angles to each other but the conformations of the two independent molecules are not identical. The spiro[4,4]nonane system found here has not been observed in any other types of antibiotics.

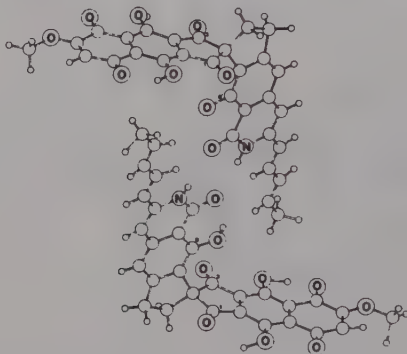
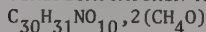


Fig. 1. The two independent molecules of fredericamycin A.

## VIRIDICATUMTOXIN METHANOL SOLVATE



J.V. SILVERTON, C. KABUTO and T. AKIYAMA, 1982. *Acta Cryst.*, **B38**, 3032-3037.

Monoclinic,  $I2$ ,  $a = 12.9784$ ,  $b = 7.8029$ ,  $c = 29.3152 \text{ \AA}$ ,  $\beta = 99.309^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.031$  for 2720 reflexions.

Work described in a preliminary communication (1) is extended by a new data collection which allowed the absolute configuration to be determined (Fig. 1).

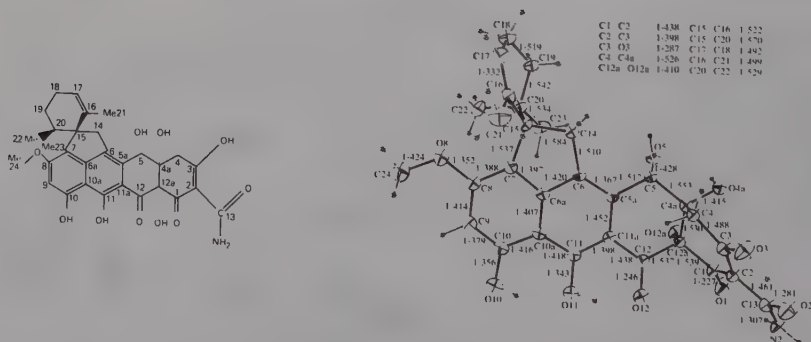
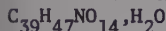


Fig. 1. The absolute configuration of viridicatumtoxin and bond lengths.

1. C. KABUTO, J.V. SILVERTON, T. AKIYAMA, U. SANKAWA, R.D. HUTCHISON, P.S. STEYN and R. VLEGGAAR, 1976. *J. Chem. Soc. Chem. Commun.*, 728-729.

## 3-METHOXYCARBONYLRIFAMYCIN S MONOHYDRATE



L. CELLAI, S. CERRINI, A. SEGRE, M. BRUFANI, W. FEDELI and A. VACIAGO, 1982. J. Chem. Soc. Perkin II, 1633-1640.

Monoclinic,  $P2_1$ ,  $a = 13.610$ ,  $b = 15.845$ ,  $c = 10.129$  Å,  $\beta = 113.37^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.047$  for 3363 reflexions.

The structure of this ansamycin antibiotic derivative (Fig. 1) is similar to a number of related compounds. The mean plane of the seventeen atom aliphatic chain makes an angle of  $97^\circ$  with the mean plane of the ten atoms of the aromatic system ( $109$  and  $98^\circ$  for other such compounds). The methoxy carbonyl substituent on C(3) is planar and at an angle of  $62^\circ$  to the aromatic rings, allowing the carbonyl and methoxy oxygens to each lie at roughly equal distances from O(11) and O(12). The torsion angles about the amide link are C(1)-C(2)-N-C(15)  $-141$  and N-C(15)-C(16)-C(17)  $63^\circ$ , resulting in an anticlinal conformation of the C(16)=C(17) bond with respect to the amide carbonyl. Intramolecular hydrogen bonds occur between O(1)...O(2) (2.54), O(8)...O(9) (2.89) and O(9)...O(10) (2.72 Å). Each water solvent molecule is involved as a donor in two intermolecular hydrogen bonds,  $H_2O...O(10)$  (2.89) and  $H_2O...O(13)$  (2.92 Å) where O(10) and O(13) belong to different molecules related by a screw axis.

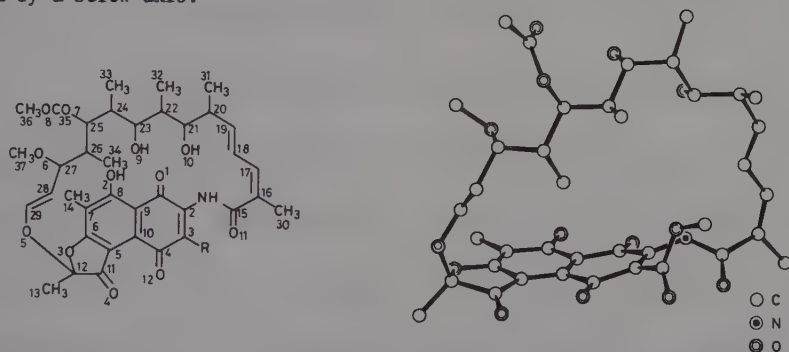
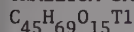


Fig. 1. Molecular skeleton ( $R = CO_2CH_3$ ) and structure of  $C_{39}H_{47}NO_{14} \cdot H_2O$ .

## THALLIUM SALT OF CATIONOMYCIN



T. SAKURAI, K. KOBAYASHI, G. NAKAMURA and K. ISONO, 1982. Acta Cryst., B38, 2471-2473.

Orthorhombic,  $P2_12_12_1$ ,  $a = 21.194$ ,  $b = 16.486$ ,  $c = 14.130$  Å,  $Z = 4$ . Mo radiation,  $R = 0.058$  for 3099 reflexions.

The molecule (Fig. 1) has a tadpole shape with the head surrounding the Tl atom which is coordinated by seven O atoms with Tl...O distances 2.79(1) to 3.07(1) Å. The exterior surface of the head consists of hydrophobic groups.

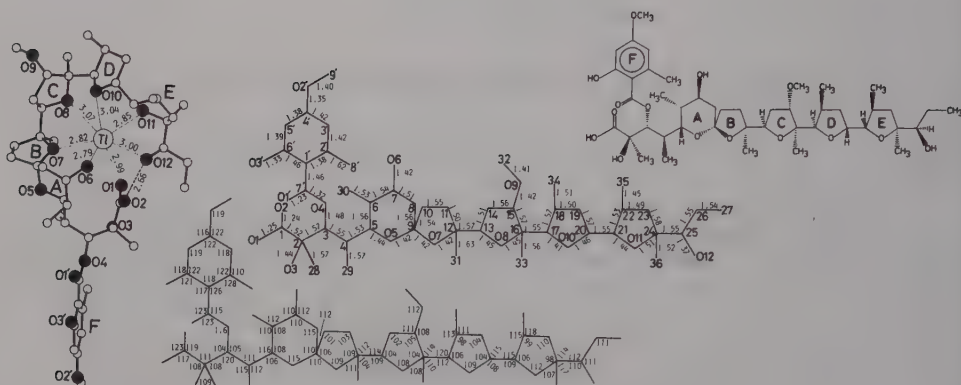
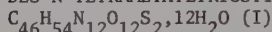
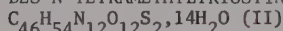


Fig. 1.  $C_{45}H_{69}O_{15}Tl$ : molecular shape and bond lengths and angles.

DES-N-TETRAMETHYLTRIOSTIN A DODECAHYDRATE (TANDEM DODECAHYDRATE)



DES-N-TETRAMETHYLTRIOSTIN A TETRADECAHYDRATE (TANDEM TETRADECAHYDRATE)



M.B. HOSSAIN, D. van der HELM, R.K. OLSEN, P.G. JONES, G.M. SHELDRICK, E. EGERT, O. KENNARD, M.J. WARING and M.A. VISWAMITRA, 1982. J. Am. Chem. Soc., 104, 3401-3408.

I. Orthorhombic,  $P2_12_12_1$ ,  $a = 18.400$ ,  $b = 22.126$ ,  $c = 16.435$  Å,  $Z = 4$ . Mo radiation,  $R = 0.147$  for 4111 reflexions at  $7^\circ C$ .

II. Orthorhombic,  $P2_12_12_1$ ,  $a = 17.989$ ,  $b = 21.793$ ,  $c = 16.135$  Å,  $Z = 4$ . Cu radiation,  $R = 0.088$  for 5192 reflexions at  $-135^\circ C$ .

The molecule (Fig. 1) has approximate twofold symmetry, with the quinoxaline chromophores and the disulphide cross-bridge projecting from opposite sides of the peptide ring. The peptide backbone resembles a distorted antiparallel  $\beta$  ribbon joined by intramolecular H-bonds  $N-H(L-Val) \dots O(L-Ala)$ . At  $-135^\circ C$  the molecule is surrounded by a regular 1st- and 2nd-order hydration sphere of 14  $H_2O$  molecules. At  $7^\circ C$  only the 1st order hydration shell is maintained. Bond lengths are as expected.

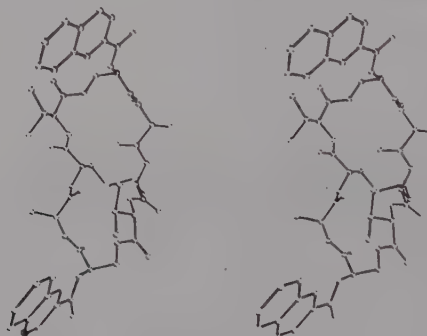
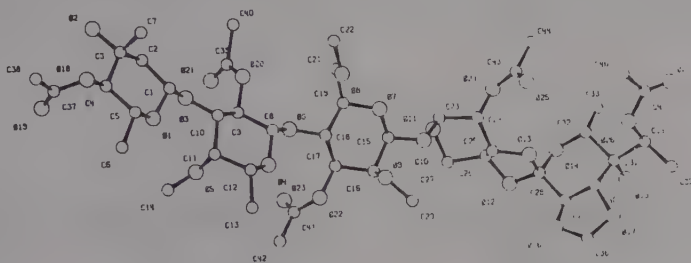


Fig. 1. The des-N-tetramethyltriostin molecule at  $-135^\circ C$ .

## PENTA-O-ACETYL-AVILEUREKANOSE ACETONE HYDRATE

 $C_{46}H_{70}O_{27} \cdot 2.35(C_3H_6O), 2H_2O$ E. KUPFER, K. NEUPERT-LAVES, M. DUBLER and W. KELLER-SCHIERLEIN, 1982. *Helv. Chim. Acta*, **65**, 3-12.Triclinic,  $P1$ ,  $a = 9.049$ ,  $b = 13.609$ ,  $c = 13.650$  Å,  $\alpha = 72.79$ ,  $\beta = 86.37$ ,  $\gamma = 85.21^\circ$ ,  $D_m = 1.258$ ,  $Z = 1$ . Mo radiation,  $R = 0.084$  for 2959 reflexions.

The analysis established the structure of this derivative of a product of alkaline hydrolysis of orthosomycin antibiotics to be as shown in Fig. 1.

Fig. 1. The  $C_{46}H_{70}O_{27}$  molecule.

## ELAIOPHYLIN METHANOL SOLVATE

 $C_{54}H_{88}O_{18} \cdot 2(CH_4O)$ K. NEUPERT-LAVES and M. DOBLER, 1982. *Helv. Chim. Acta*, **65**, 262-267.Monoclinic,  $P2_1$ ,  $a = 9.927$ ,  $b = 10.105$ ,  $c = 31.183$  Å,  $\beta = 93.20^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.041$  for 3182 reflexions.

The analysis confirms the constitution of elaiophylin (Fig. 1). The two chemically equivalent halves of the molecule are related by an approximate twofold axis. Four intramolecular O-H...O hydrogen bonds are formed utilising hydroxyls 04, 04', 05 and 05' to oxygens 02, 02', 04 and 04' respectively with O...O 2.708-3.089 Å.

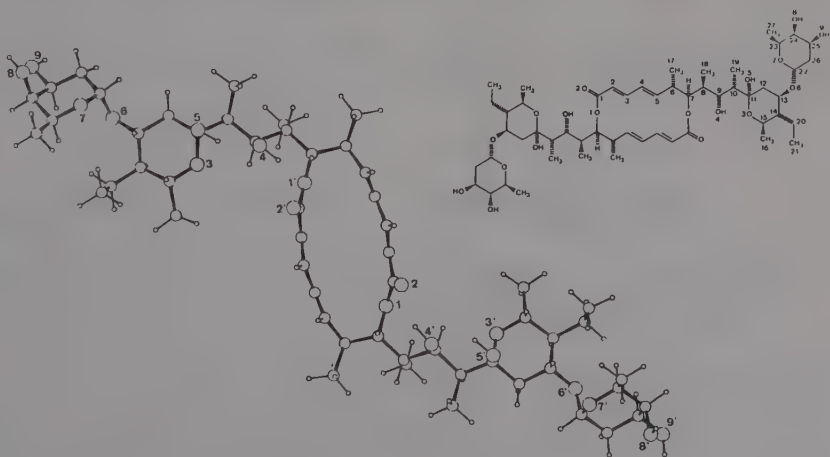


Fig. 1. The elaiophylin molecule.

VALINOMYCIN SODIUM PICRATE MONOHYDRATE *m*-XYLENE SOLVATE
$$\text{C}_{60}\text{H}_{94}\text{N}_9\text{NaO}_{26}, \text{C}_8\text{H}_{11} (\text{C}_{54}\text{H}_{90}\text{N}_6\text{O}_{18})\text{Na}^+ (\text{C}_6\text{H}_2\text{N}_3\text{O}_7)^- (\text{H}_2\text{O}), \text{C}_8\text{H}_{11}$$

L.K. STEINRAUF, J.A. HAMILTON and M.N. SABESAN, 1982. J. Am. Chem. Soc., **104**, 4085-4091.

Monoclinic,  $P2_1$ ,  $a = 13.369$ ,  $b = 21.908$ ,  $c = 15.818$  Å,  $\beta = 122.1^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.15$  for 3582 reflexions (at  $-170^\circ\text{C}$ ).

The structure (Fig. 1) revealed a water molecule in the region within valinomycin usually occupied by a potassium ion (1). The  $\text{Na}^+$  ion was found to be external; bonding to three carbonyl O atoms adjacent to the lactyl residues, the water molecule, and to two picrate O atoms (Na-O 2.33(1)-2.64(2) Å).

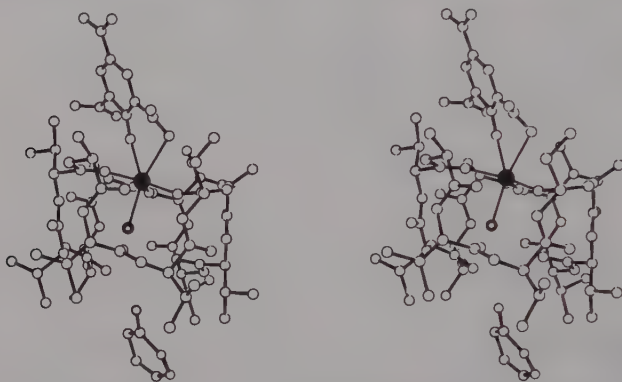


Fig. 1. Stereoview of valinomycin sodium picrate monohydrate *m*-xylene solvate.

1. Structure Reports, **48B**, 770.

14 $\alpha$ ,17 $\beta$ -DIHYDROXY-5-METHOXY-DE-A-OESTRA-5,7,9-TRIEN-15-ONE MONOHYDRATE
$$\text{C}_{15}\text{H}_{18}\text{O}_4 \cdot \text{H}_2\text{O}$$

P.J. COX, R.A. HOWIE, A.W. NOWICKI and A.B. TURNER, 1982. J. Chem. Soc. Perkin I, 657-664.

Monoclinic,  $P2_1$ ,  $a = 8.320$ ,  $b = 7.026$ ,  $c = 12.816$  Å,  $\beta = 110.11^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.069$  for 1216 reflexions.

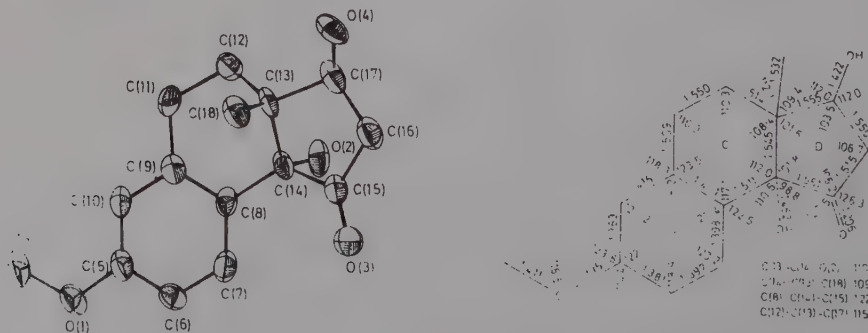
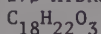


Fig. 1. The molecular structure, bond lengths and angles in  $\text{C}_{15}\text{H}_{18}\text{O}_4 \cdot \text{H}_2\text{O}$ .

The molecular structure and the bond distances and angles are shown in Fig. 1. The C/D ring junction is trans. Ring C adopts a sofa conformation and the conformation of ring D is midway between envelope and half chair, similar to other steroids with aromatic rings and trans C/D ring junctions. There are angular distortions around C(14), C(8)-C(14)-C(15) 122.1(6) and C(15)-C(14)-O(2) 98.8(4)° being respectively larger and smaller than the tetrahedral angle. The O(3)...H(7) separation is 2.40 Å and the dihedral angle between the aromatic ring B and the carbonyl atoms C(14), C(15), C(16), and O(3) is 18.3°. The crystal is a monohydrate and the molecules are linked by hydrogen bonding.

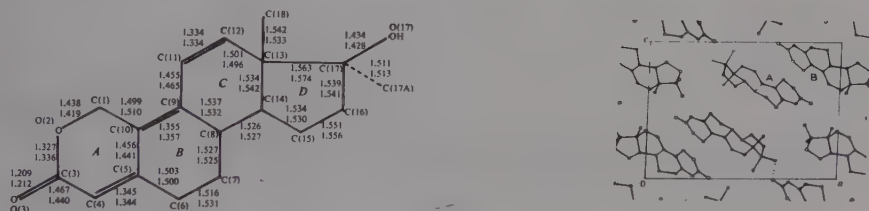
17 $\beta$ -HYDROXY-17 $\alpha$ -METHYL-2-OXAESTRATRI-4,9,11-ENE-3-ONE (FORM 1)



G. LEPICARD, J. DELETTRE and J.-P. MORNON, 1982. *Acta Cryst.*, B38, 2968-2970.

Monoclinic,  $P2_1$ ,  $a = 17.875$ ,  $b = 6.653$ ,  $c = 12.929$  Å,  $\beta = 92.15^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.055$  for 3220 reflexions.

In the crystal structure (Fig. 1) the two independent molecules are linked into chains by O-H...O hydrogen bonds with O...O 2.94(2) and 2.86(2) Å. For the crystal structure of Form 2 see (1).





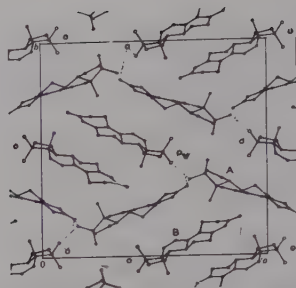
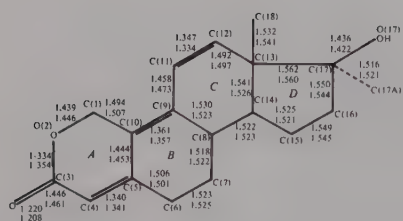


Fig. 1.  $C_{18}H_{22}O_3 \cdot H_2O$ : bond lengths in the two independent molecules (upper and lower values) and a view of the crystal structure projected down c.

# 1. Preceding Report.

## 4-FLUORO-1,3,5(10)-ESTRATRIENE-3,17 $\beta$ -DIOL HEMIMETHANOL $C_{18}H_{23}FO_2 \cdot 0.5(CH_4O)$

K. GO, G. KARTHA and M. NEEMAN, 1982. Acta Cryst., B38, 3142-3144.

Triclinic,  $P1$ ,  $a = 7.367$ ,  $b = 9.363$ ,  $c = 12.531$  Å,  $\alpha = 89.31^\circ$ ,  $\beta = 93.38^\circ$ ,  $\gamma = 109.62^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.062$  for 3043 reflexions.

There are two independent molecules in the unit cell (Fig. 1) with essentially similar conformations; the A ring is planar, the B ring is 7 $\alpha$ ,8 $\beta$ -half-chair, the C ring is a chair and the D ring between a 13 $\beta$ -envelope 13 $\beta$ ,14 $\alpha$ -half-chair.

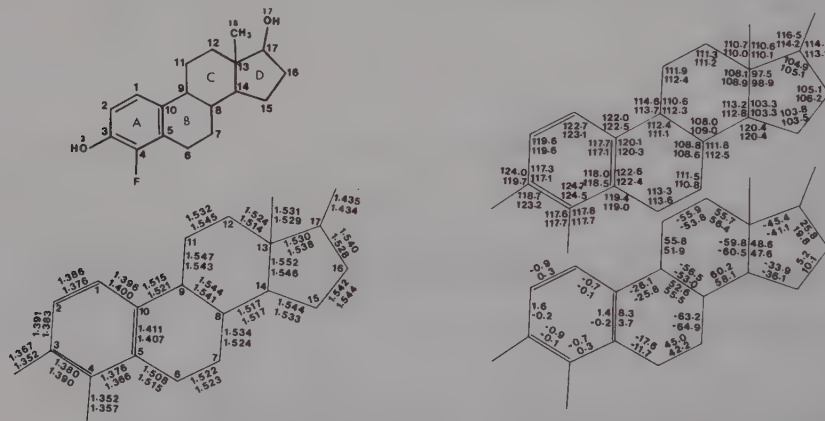


Fig. 1. The  $C_{18}H_{23}FO_2$  molecule and dimensions.

## 17 $\beta$ -HYDROXY-2-AZA-4-ANDROSTEN-3-ONE $C_{18}H_{27}NO_2$

J.L. BRIANSO, M.C. BRIANSO and J.F. PINIELLA, 1982. Cryst. Struct. Comm., 11, 95-99.

Monoclinic,  $P2_1$ ,  $a = 10.093$ ,  $b = 10.551$ ,  $c = 7.690$  Å,  $\gamma = 90.55^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.08$  for 1254 reflexions.

The bond distances and angles are in the normally observed ranges for this type of molecule. The B and C rings (Fig. 1) have normal chair conformations and the D

ring has  $14\alpha$ -envelope conformation. In A ring the bond distances N(2)-C(3), C(3)-O(20) and C(4)-C(5) suggest that this  $\alpha,\beta$ -unsaturated lactam is partially diene in character in agreement with lactim canonical form.

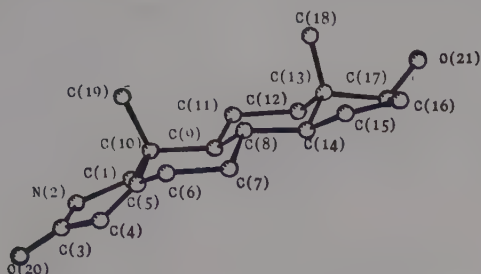


Fig. 1. A view showing the conformation of  $C_{18}H_{27}NO_2$ .

16 $\beta$ -BROMO-3 $\beta$ -HYDROXY-5-ANDROSTEN-17-ONE

$C_{19}H_{27}BrO_2$

D.C. SWENSON, W.L. DUAX, Y. OSAWA and M. NUMAZAWA, 1982. Cryst. Struct. Comm., 11, 701-706.

Monoclinic,  $P2_1$ ,  $a = 12.176$ ,  $b = 7.989$ ,  $c = 9.464$  Å,  $\beta = 104.29^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.097$  for 1392 reflexions.

The A and C rings (Fig. 1) have normal chair conformations, ring B is a  $8\beta,9\alpha$  half-chair and ring D is a  $14\alpha$  envelope. Molecules are linked by O-H...O hydrogen bonds ( $O\cdots O$  2.90 Å). The molecule is compared with the 16 $\alpha$ -bromo isomer (1).

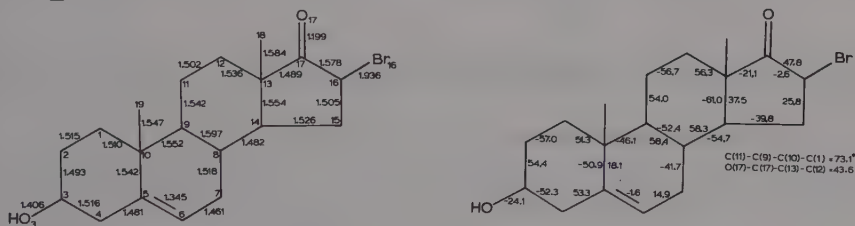


Fig. 1. Bond lengths and torsion angles in  $C_{19}H_{27}BrO_2$ .

1. Structure Reports, 41B, 594.

11 $\beta$ ,17 $\beta$ -DIHYDROXY-19-NORPREGNA-4,9-DIENE-20-YNE-3-ONE

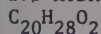
$C_{20}H_{24}O_3$

G. LEPICARD, J.P. MORNON and J. DELETTRE, 1982. Acta Cryst., B38, 1644-1645.

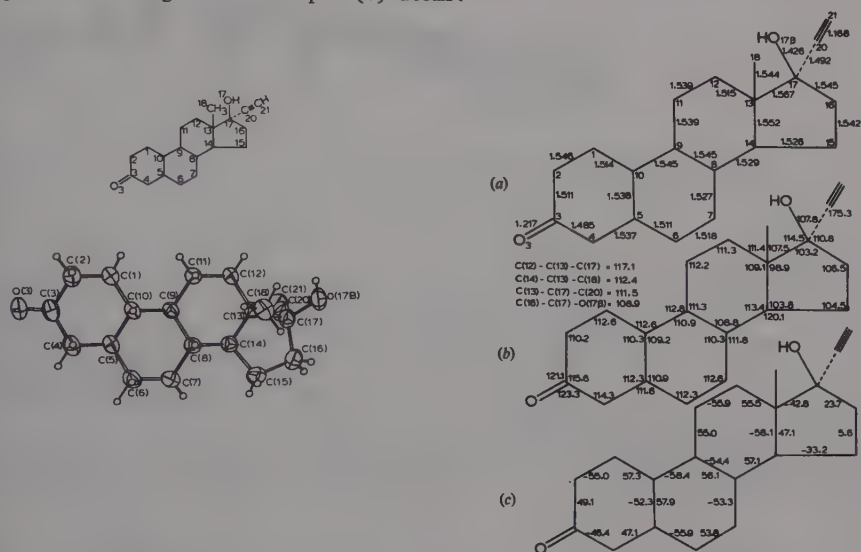
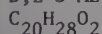
Orthorhombic,  $P2_12_12_1$ ,  $a = 17.76$ ,  $b = 11.78$ ,  $c = 7.570$ ,  $Z = 4$ . Mo radiation,  $R = 0.063$  for 2218 reflexions.

The steroid skeleton is almost planar, with the 11 $\beta$ -OH group in an uncommon orientation. Ring A (Fig. 1) has a half-chair conformation. Molecules are linked by hydrogen bonds ( $O(17)\cdots O(11)$  2.94,  $O(11)\cdots O(3)$  2.75 Å).



17 $\beta$ -HYDROXY-19-NOR-5 $\alpha$ ,17 $\alpha$ -PREGN-20-YN-3-ONED.C. ROHRER, W.L. DUAX, R. PETERS and M. TANABE, 1982. *Acta Cryst.*, **B38**, 1362-1364.Orthorhombic,  $P2_12_12_1$ ,  $a = 12.391$ ,  $b = 21.066$ ,  $c = 6.597$  Å,  $Z = 4$ . Mo radiation,  $R = 0.063$  for 1477 reflexions.

In the molecule (Fig. 1) rings A, B and C all have a chair conformation and ring D has a distorted C(13)  $\beta$ -envelope conformation. The ring A chair is slightly flattened in the region of the carbonyl substituent in order to accommodate the larger valence angles of the  $sp^2$  C(3) atoms.

Fig. 1.  $C_{20}H_{28}O_2$ : a perspective view of the molecule and dimensions.D,L-3-METHOXY-D-HOMO-8 $\alpha$ -ESTRA-1,3,5(10)-TRIEN-17 $\alpha\beta$ -OLA.N. CHEKHOV, S.P. IONOV, V.V. EGOROVA and S.N. ANANCHENKO, 1982. *Cryst. Struct. Comm.*, **11**, 675-680.Monoclinic,  $P2_1/n$ ,  $a = 12.547$ ,  $b = 6.137$ ,  $c = 21.677$  Å,  $\beta = 91.55^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.0262$  for 1285 reflexions.

In the molecule (Fig. 1), ring A is planar, ring B is a 7 $\beta$ ,8 $\alpha$  half-chair, and rings C and D are normal chairs. Molecules are linked by O-H...O hydrogen bonds (2.830(2) Å).

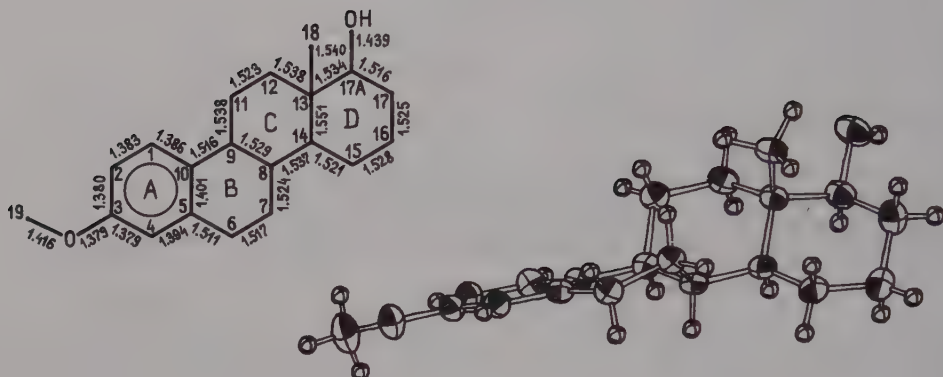


Fig. 1. A view of  $C_{20}H_{28}O_2$  and bond lengths.

7 $\alpha$ -METHYL-4-ANDROSTENE-3,17-DIONE



H.J. DE L. COWE, P.J. COX and G.A. SIM, 1982. *Acta Cryst.*, **B38**, 662-664.

Orthorhombic,  $P2_12_12_1$ ,  $a = 6.424$ ,  $b = 9.466$ ,  $c = 27.008$  Å,  $D_m = 1.21$ ,  $Z = 4$ . Mo radiation,  $R = 0.038$  for 1452 reflexions.

In the molecule (Fig. 1) the conformations of the four rings are; A, 1 $\alpha$ -sofa; B and C, chair; D, envelope with C(14) at the flap. The molecular geometry is normal.

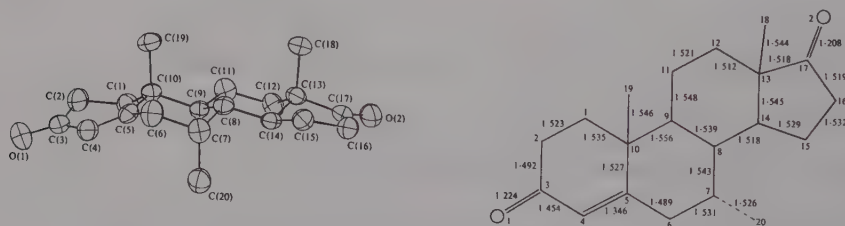


Fig. 1. The 7 $\alpha$ -methyl-4-androstene-3,17-dione molecule.

A-NOR-PREGN-3-EN-2,20-DIONE



V. BERTOLASI and J.-P. MORNON, 1982. *Cryst. Struct. Comm.*, **11**, 509-515.

Orthorhombic,  $P2_12_12_1$ ,  $a = 24.033$ ,  $b = 12.359$ ,  $c = 11.360$  Å,  $Z = 8$ . Cu radiation,  $R = 0.052$  for 2530 reflexions.

The crystal packing is shown in Fig. 1. Bond lengths and angles in the two independent molecules are normal. Intermolecular contacts correspond to van der Waals interactions.

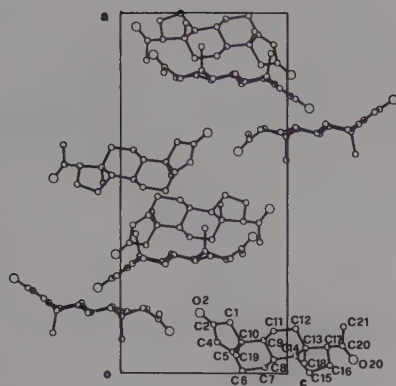


Fig. 1. The packing of  $C_{20}H_{28}O_2$ .

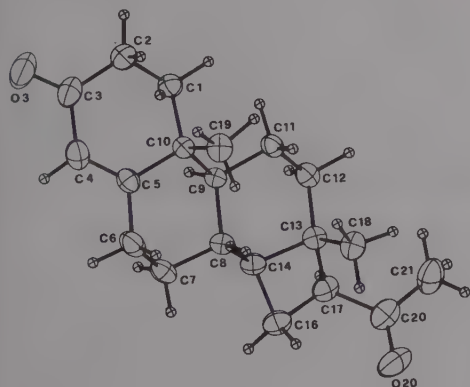
D-NOR-PREGN-4-EN-3,20-DIONE

$C_{20}H_{28}O_2$

V. BERTOLASI and J.-P. MORNON, 1982. Cryst. Struct. Comm., **11**, 517-522.

Tetragonal,  $P4_1$ ,  $a = 8.405$ ,  $c = 24.432 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.034$  for 1362 reflexions.

Bond lengths and angles are as expected. The conformation and some torsion angles are shown in Fig. 1. Intermolecular contacts correspond to van der Waals interactions.



C(10)-C(1)-C(2)-C(3)	-57.7(4)
C(1)-C(2)-C(3)-C(4)	38.1(5)
C(2)-C(3)-C(4)-C(5)	-7.8(7)
C(3)-C(4)-C(5)-C(10)	-6.2(7)
C(4)-C(5)-C(10)-C(1)	-12.2(5)
C(2)-C(1)-C(10)-C(5)	44.0(4)
C(10)-C(5)-C(6)-C(7)	-53.1(4)
C(5)-C(6)-C(7)-C(8)	55.3(4)
C(6)-C(7)-C(8)-C(9)	-57.6(4)
C(7)-C(8)-C(9)-C(10)	57.6(4)
C(8)-C(9)-C(10)-C(5)	-50.6(4)
C(6)-C(5)-C(10)-C(9)	49.4(4)
C(8)-C(9)-C(11)-C(12)	47.0(4)
C(9)-C(11)-C(12)-C(13)	-50.7(4)
C(11)-C(12)-C(13)-C(14)	59.5(4)
C(12)-C(13)-C(14)-C(8)	-72.5(4)
C(9)-C(8)-C(14)-C(13)	63.2(4)
C(13)-C(14)-C(16)-C(17)	-24.9(3)
C(14)-C(16)-C(17)-C(13)	24.2(3)
C(14)-C(13)-C(17)-C(16)	-24.5(3)
C(17)-C(13)-C(14)-C(16)	24.3(3)
C(16)-C(17)-C(20)-O(20)	8.4(6)

Fig. 1. The D-nor-pregn-4-en-3,20-dione molecule.

3 $\alpha$ -ACETOXY-7 $\alpha$ ,15 $\alpha$ -OXIDO-16-OXA-5 $\beta$ ,14 $\beta$ -ANDROSTAN-17-ONE

$C_{20}H_{28}O_5$

E.O. SCHLEMPER and B. NASSIM, 1982. Cryst. Struct. Comm., **11**, 2013-2019.

Monoclinic,  $P2_1$ ,  $a = 12.590$ ,  $b = 12.336$ ,  $c = 12.760 \text{ \AA}$ ,  $\beta = 113.77^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.065$  for 1832 reflexions.



The two independent molecules (Fig. 1) differ only in the orientation of the acetate group ( $C2-C3-OAC1-CAC1$   $-84.6^\circ$  in one molecule and  $-149.0$  in the other).

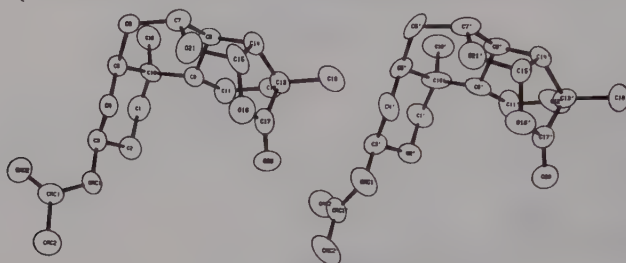


Fig. 1. Views of the two  $C_{20}H_{28}O_5$  molecules.

17 $\beta$ -HYDROXY-7 $\alpha$ -METHYL-4-ANDROSTEN-3-ONE (7 $\alpha$ -METHYLTESTOSTERONE)  
 $C_{20}H_{30}O_2$

P.J. COX and G.A. SIM, 1982. Acta Cryst., B38, 1360-1362.

Monoclinic,  $P2_1$ ,  $a = 11.016$ ,  $b = 6.156$ ,  $c = 12.967$  Å,  $\beta = 104.49^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.045$  for 1420 reflexions.

In the molecule (Fig. 1) rings B and C are observed in chair conformations, ring A adopts a  $1\alpha$ -sofa conformation and ring D a conformation midway between a C(13)-envelope and a half-chair. An O(2)-H...O(1) hydrogen bond (2.832 Å) provides head-to-tail linkage between molecules in the crystal.

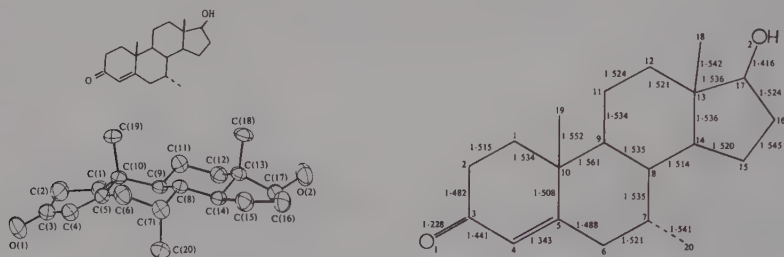


Fig. 1.  $C_{20}H_{30}O_2$ : a perspective view of the molecule and bond lengths.

17-METHOXYCARBONYL-15,16-DIAZA-15-PHENYL-7,11-DITHIAGONA-1,3,5(10),8,13,16-HEXAENE  
 $C_{21}H_{16}N_2O_2S_2$

G.V.N.A. RAO, S.R. RAMADAS and M.V. KRISHNA, 1982. Cryst. Struct. Comm., 11, 1799-1804.

Monoclinic,  $P2_1/c$ ,  $a = 14.048$ ,  $b = 9.179$ ,  $c = 15.083$  Å,  $\beta = 108.624^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.033$  for 1719 reflexions.

The analysis establishes the structure of this reaction product to be as shown in Fig. 1. Some bond lengths are: N-N 1.359(5), S-C( $sp^3$ ) 1.815(5), S-C( $sp^2$ ) 1.767(4) Å.

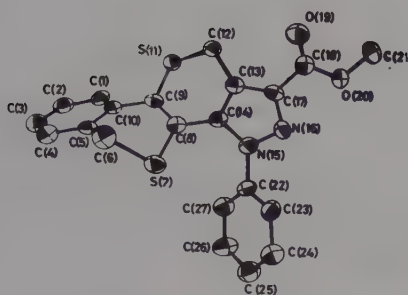


Fig. 1. The  $C_{21}H_{16}N_2O_2S_2$  molecule.

13 $\beta$ -ETHYL-17 $\beta$ -HYDROXY-18,19-DINOR-17 $\alpha$ -PREGNATRI-4,9,11-ENE-20-YNE-3-ONE TOLUENE  
 $C_{21}H_{24}O_2 \cdot 0.5(C_7H_8)$

J. DELETTRE, G. LEPICARD and J.-P. MORNON, 1982. *Acta Cryst.*, B38, 2965-2968.

Monoclinic,  $P2_1$ ,  $a = 7.513$ ,  $b = 21.150$ ,  $c = 12.745$  Å,  $\beta = 100.37^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.084$  for 3100 reflexions.

In the crystal structure (Fig. 1) the toluene molecule is in close contact with the B rings of the two independent steroid molecules and possesses a higher thermal vibration than that of the steroid molecules.

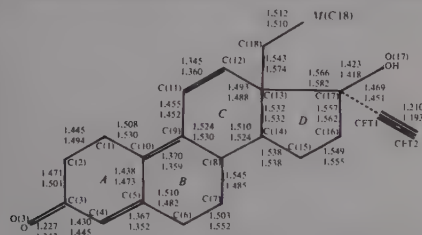
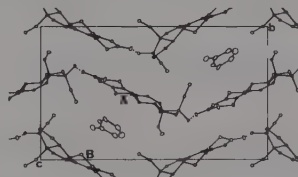


Fig. 1.  $(C_{21}H_{24}O_2)_2 \cdot C_7H_8$ : bond lengths in the two independent steroid molecules (upper and lower values) and a view of the a-axial projection of the crystal structure.



2 $\alpha$ ,17 $\alpha$ -BIS(2-CHLOROETHYNYL)-A-nor-5 $\alpha$ -OESTRANE-2 $\beta$ ,17 $\beta$ -DIOL METHANOL  
 $C_{21}H_{26}Cl_2O_2 \cdot 2(CH_4O)$

F. BAERT, M. FOULON and G. ODOU, 1982. *Acta Cryst.*, B38, 3139-3142.

Triclinic,  $P1$ ,  $a = 8.438$ ,  $b = 10.339$ ,  $c = 7.155$  Å,  $\alpha = 98.37^\circ$ ,  $\beta = 92.83^\circ$ ,  $\gamma = 97.02^\circ$ ,  $Z = 1$ .  $R = 0.049$  for 1896 reflexions.

Bond lengths are close to accepted values but the molecule is significantly bent (Fig. 1).

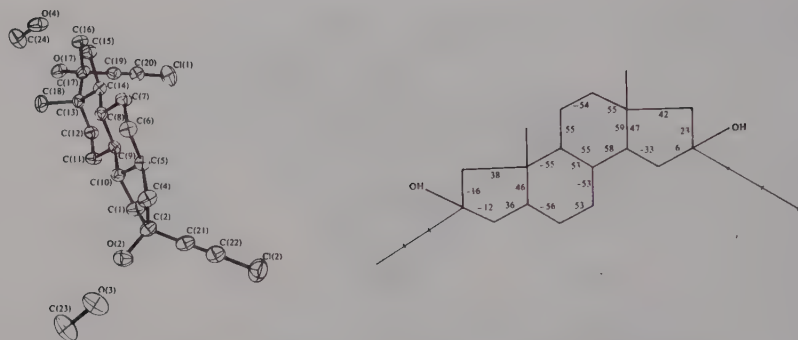


Fig. 1. The  $C_{21}H_{26}Cl_2O_2, 2(CH_4O)$  structure and ring torsion angles.

11 $\beta$ , 17 $\beta$ -DIHYDROXY-18-METHYL-19-NORPREGNA-4, 9-DIENE-20-YNE-3-ONE

$C_{21}H_{26}O_3$

J.P. MORNON, J. DELETTRE and G. LEPICARD, 1982. Acta Cryst., B38, 1638-1641.

Orthorhombic,  $P2_12_12_1$ ,  $a = 24.85$ ,  $b = 16.651$ ,  $c = 8.736 \text{ \AA}$ ,  $Z = 8$ . Mo radiation,  $R = 0.134$  for 782 reflexions.

The structure is isomorphous with a previously found one of the same steroid (1) but one molecule is disordered between two very different positions each associated with a particular hydrogen bond. There are also ethynyl...oxygen contacts of length 3.35 and 3.52  $\text{\AA}$ .

1. Structure Reports, 43B, 663.

( $\pm$ )-3-METHOXY-17 $\alpha$ -ACETOXY-16-THIA-D-HOMOEOSTRA-1,3,5(10),8-TETRAENE

$C_{21}H_{26}O_3S$

M. SHIRO, H. NAKAI, T. OKADA and T. TERASAWA, 1982. Cryst. Struct. Comm., 11, 423-426.

Triclinic,  $P\bar{1}$ ,  $a = 11.337$ ,  $b = 13.134$ ,  $c = 7.656 \text{ \AA}$ ,  $\alpha = 105.44$ ,  $\beta = 117.32$ ,  $\gamma = 68.11^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.050$  for 2922 reflexions.

The analysis establishes the  $\alpha$ -configuration of hydrogen atoms at C14 and C17a (Fig. 1).

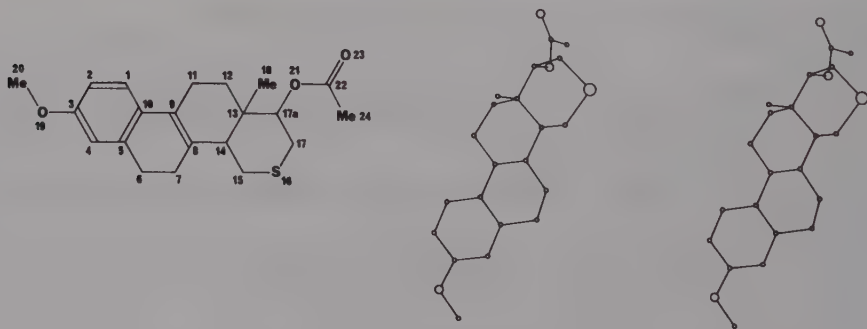
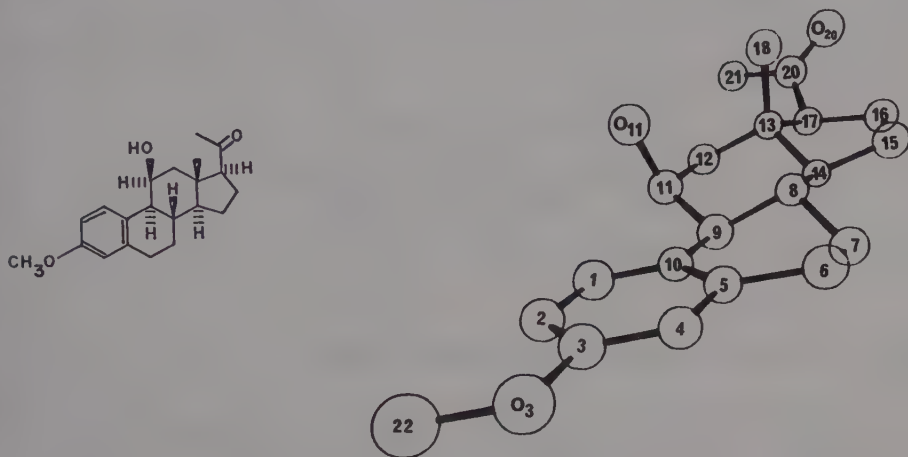


Fig. 1. The  $C_{21}H_{26}O_3S$  molecule and a stereoview.

11 $\beta$ -HYDROXY-3-METHOXY-19-NORPREGNA-1,3,5(10)-TRIENE-20-ONE $C_{21}H_{28}O_3$ K.-H. CHAO, T.-F. WANG and F.E. ZIEGLER, 1982. Cryst. Struct. Comm., 11, 965-970.Monoclinic,  $P2_1/n$ ,  $a = 13.078$ ,  $b = 6.414$ ,  $c = 22.169$  Å,  $\beta = 110.06^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.073$  for 1093 reflexions.

In this major component of a cyclization reaction, the conformation at all chiral centres is shown to be the desired stereochemistry; that is, trans-anti-trans geometry on the ring junctures, 11 $\beta$ -hydroxy group and 17 $\beta$ -acetyl group (Fig. 1).

Fig. 1. The  $C_{21}H_{28}O_3$  molecule.

## 18-DEOXYALDOSTERONE HEMIHYDRATE

 $C_{21}H_{28}O_4 \cdot 0.5(H_2O)$ W.L. DUAX, J.F. GRIFFIN, P.D. STRONG, J.W. FUNDER and S. ULICK, 1982. J. Am. Chem. Soc., 104, 7291-7293.Orthorhombic,  $P2_12_12_1$ ,  $a = 19.878$ ,  $b = 30.341$ ,  $c = 5.9951$  Å,  $Z = 8$ . Cu radiation,  $R = 0.072$  for 2049 reflexions.

The structure contains two independent molecules which differ significantly in 17 $\beta$ -side-chain orientation, 4-en-3-one conjugation, and hydrogen bonding (Fig. 1). The C(20)-O(20) bond is synperiplanar to C(17)-C(16) in molecule I and to C(17)-C(13) in molecule II. The latter conformation, previously observed only in the presence of a 16 $\beta$ -methyl or -halo substituent, has been stabilized by the fact that the epoxide formation draws the C(18) methyl away from the D ring. Both molecules have similar H-bonding to O(3) and nearly planar 4-en-3-one conformations.

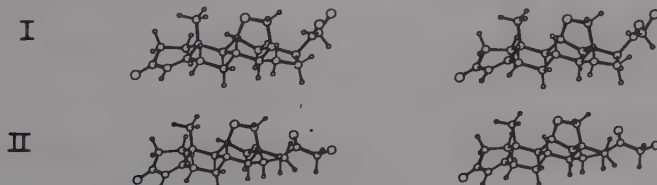


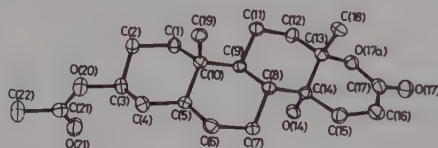
Fig. 1. Stereoviews of the two independent molecules of 18-deoxyaldosterone.

3 $\beta$ -ACETOXY-14 $\alpha$ -HYDROXY-17 $\alpha$ -OXA-D-HOMO-5 $\alpha$ -ANDROST-15-EN-17-ONE $C_{21}H_{30}O_5$ 

D.H.R. BARTON, R.A.H.F. HUI, S.V. LEY and D.J. WILLIAMS, 1982. J. Chem. Soc. Perkin I, 1919-1922.

Orthorhombic,  $P2_12_12_1$ ,  $a = 14.435$ ,  $b = 20.896$ ,  $c = 6.469$  Å,  $Z = 4$ . Cu radiation,  $R = 0.051$  for 1547 reflexions.

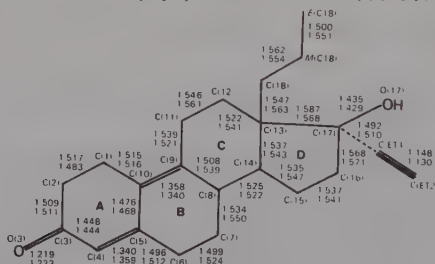
The carbonyl group of the unsaturated lactone (Fig. 1) is not coplanar with the adjacent double bond, the C(15)-C(16)-C(17)-O(17) torsion angle being  $-163.4^\circ$ . The bond angles around the lactone are C(15)-C(16)-C(17)  $122.0$ , C(16)-C(17)-O(17a)  $116.8$ , C(17)-O(17a)-C(13)  $119.9$ , C(14)-C(13)-O(17a)  $107.9$  and C(13)-C(14)-C(15)  $106.5^\circ$ . There is an intermolecular hydrogen bond of  $2.822$  Å between the 14-hydroxyl group and the acetate of a screw related molecule.

Fig. 1. Molecular structure of  $C_{21}H_{30}O_5$ .18-ETHYL-17 $\beta$ -HYDROXY-19-NORPREGNA-4,9-DIENE-20-YNE-3-ONE $C_{22}H_{28}O_2$ 

J.-P. MORNON, G. LEPICARD and J. DELETTRE, 1982. Acta Cryst., B38, 1641-1643.

Monoclinic,  $P2_1$ ,  $a = 13.260$ ,  $b = 11.694$ ,  $c = 12.571$  Å,  $\beta = 104.82^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.080$  for 3073 reflexions.

The two independent molecules A and B (Fig. 1) are slightly different but have normal conformations for 4,9-diene steroids. Identical molecules are linked into chains by O(17)...O(3) hydrogen bonds of length  $2.77$  and  $2.84$  Å respectively. Chains of B molecules are linked by  $C\equiv C-H\cdots O$  hydrogen bonds of length  $3.33(2)$  Å and A/B chains are linked by  $C\equiv C-H\cdots C$ (ethynyl) bonds with  $H\cdots C$   $2.77(2)$  Å.

Fig. 1. Bond lengths in molecules A and B of the  $C_{22}H_{28}O_2$  structure.17 $\beta$ -CYANO-16 $\alpha$ ,17-EPOXY-3 $\beta$ -ACETOXY-5-ANDROSTENE $C_{22}H_{29}NO_3$ 

D.C. SWENSON, W.L. DAUX, M. NUMAZAWA and Y. OSAWA, 1982. Cryst. Struct. Comm., 11, 617-620.

Monoclinic,  $P2_1$ ,  $a = 10.760$ ,  $b = 5.970$ ,  $c = 16.138$  Å,  $\beta = 108.25^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.076$  for 1757 reflexions.

The A and C rings (Fig. 1) have normal chair conformations. The B-ring has an 8 $\beta$ ,9 $\alpha$  half-chair conformation and ring D is near a 14 $\alpha$ -envelope conformation.

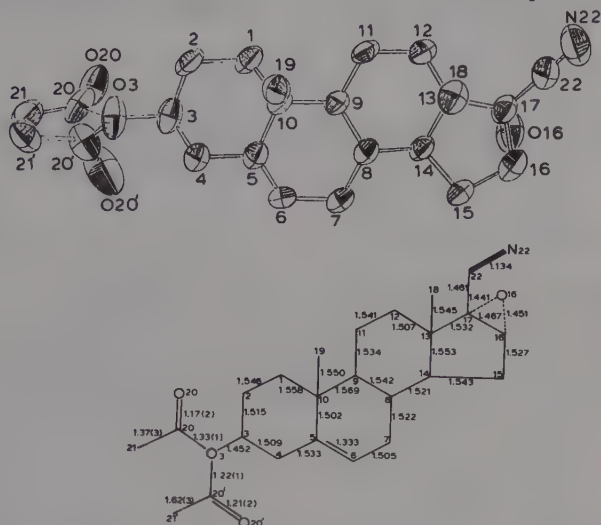


Fig. 1. The  $C_{22}H_{29}NO_3$  molecule and bond lengths.

16 $\alpha$ -ETHYL-21-FLUORO-19-NOR-4,6-PREGNADIENE-3,20-DIONE  
 $C_{22}H_{29}O_2F$

W.L. DUAX and P.D. STRONG, 1982. Cryst. Struct. Comm., 11, 1891-1894.

Orthorhombic,  $P2_12_12_1$ ,  $a = 12.068$ ,  $b = 16.846$ ,  $c = 9.484$  Å,  $Z = 4$ . Cu radiation,  $R = 0.083$  for 2140 reflexions.

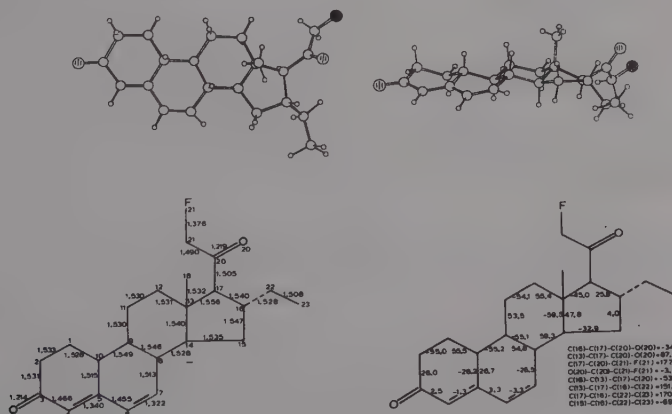


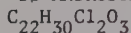
Fig. 1. The  $C_{22}H_{29}O_2F$  molecule, with bond lengths and torsion angles.

The 4,6-dien-3-one system (Fig. 1) is planar to within 0.07 Å. The A and B rings have perfect 1 $\alpha$ -sofa and 9 $\alpha$ -sofa-conformations respectively. Although the O(3)-C(3)-C(4)-C(5) angle of  $-175.2^\circ$  suggests some strain that might be relieved by A-ring conformational change to the inverted form proposed to be responsible for progesterone receptor binding, such an inversion has never been observed in a 4,6-



dien-3-one structure. The 17 $\beta$ -side chain orientation is consistent with the established pattern. The fluorine is synclinal with the 20 carbonyl and the O(20)-C(20)-C(17)-C(16) torsion angle is -34.3°. The 16 $\alpha$ -ethyl group is antiperiplanar to the C(16)-C(17) bond.

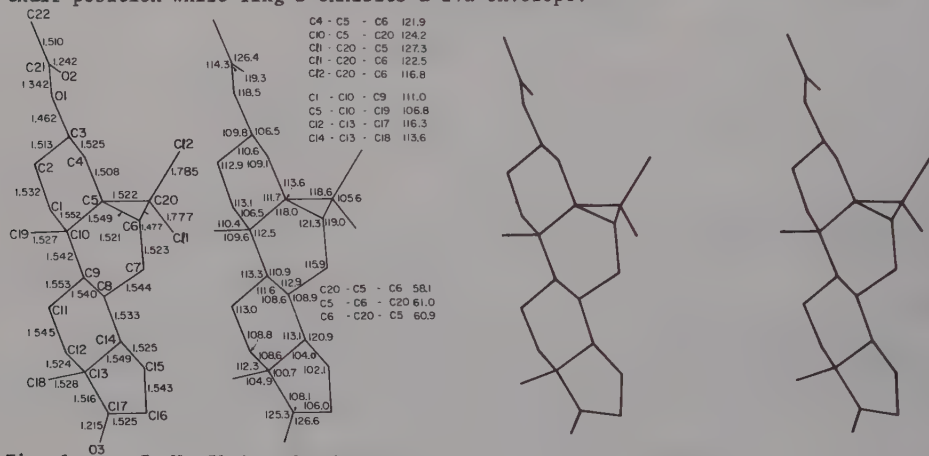
3 $\beta$ -ACETOXY-5,6 $\alpha$ -DICHLOROMETHYLENE-5 $\beta$ -ANDROSTAN-17-ONE (5,6 $\alpha$ -DICHLOROMETHYLENE-17-OXO-5 $\beta$ -ANDROSTAN-3 $\beta$ -YL ACETATE)



E.J. GABE, F.L. LEE and S.M. BOUDREAU, 1982. *Acta Cryst.*, **B38**, 2975-2977.

Orthorhombic, P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, a = 12.0931, b = 11.2148, c = 15.2104 Å, Z = 4. Mo radiation, R = 0.052 for 2640 reflexions.

In the molecule (Fig. 1) rings A and B have distorted chair conformations, both attributable to the strain of the cyclopropyl ring. Ring C adopts a normal chair position while ring D exhibits a 14 $\alpha$ -envelope.



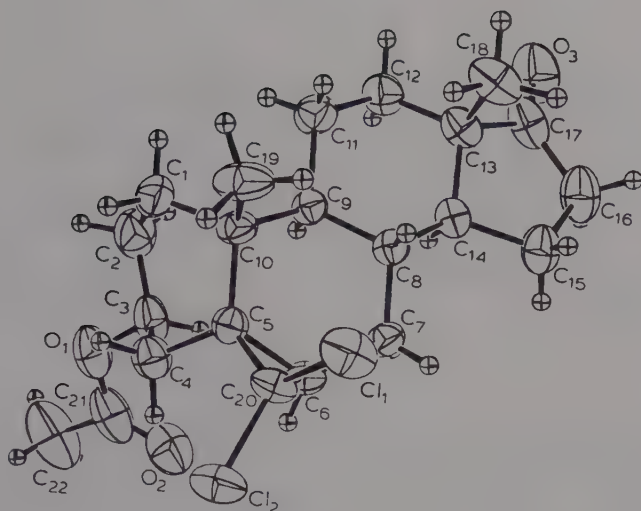


Fig. 1. A view of  $C_{22}H_{30}Cl_2O_3$ .

2α,16α-DIETHYNYL-5α-NOR-A-ANDROSTANE-2β,16β-DIOL  
 $C_{22}H_{30}O_2$

J.-P. MORNON, G. LEPICARD, J. DELETTRE, R. BALLY and C. BRASSY, 1982. *Acta Cryst.*, B38, 2973-2975.

Monoclinic,  $P2_1$ ,  $a = 23.018$ ,  $b = 5.981$ ,  $c = 6.871$  Å,  $\beta = 94.70^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.037$  for 1682 reflexions.

The observed conformation of the molecule (Fig. 1) is close to the expected one, this steroid being rigid. The molecules are linked together in the crystal by long hydrogen bonds  $O(2)-H...O(2')$  ( $O...O$  3.24 Å) and  $O(16)-H...O(16')$  ( $O...O$  3.20 Å).

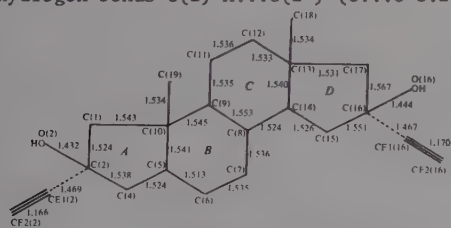
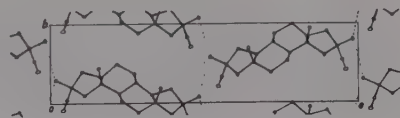


Fig. 1.  $C_{22}H_{30}O_2$ : bond lengths in the molecule and the c-axial projection of the crystal structure.

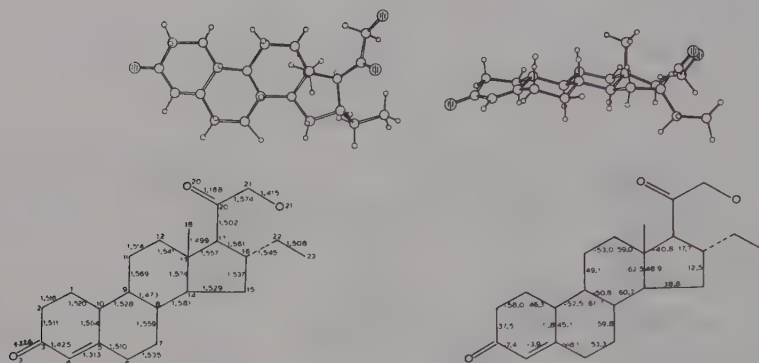


16α-ETHYL-21-HYDROXY-19-NOR-4-PREGNENE-3,20-DIONE  
 $C_{22}H_{32}O_3$

W.L. DUAX and P.D. STRONG, 1982. *Cryst. Struct. Comm.*, 11, 1885-1889.

Orthorhombic,  $P2_12_12_1$ ,  $a = 10.360$ ,  $b = 23.704$ ,  $c = 7.8691$  Å,  $Z = 4$ . Cu radiation,  $R = 0.087$  for 1487 reflexions.

In this molecule (Fig. 1) ring A is intermediate between a  $1\alpha,2\beta$ -half-chair and a  $1\alpha$ -sofa, rings B and C have normal chair form, and ring D is a  $13\beta,14\alpha$ -half-chair.



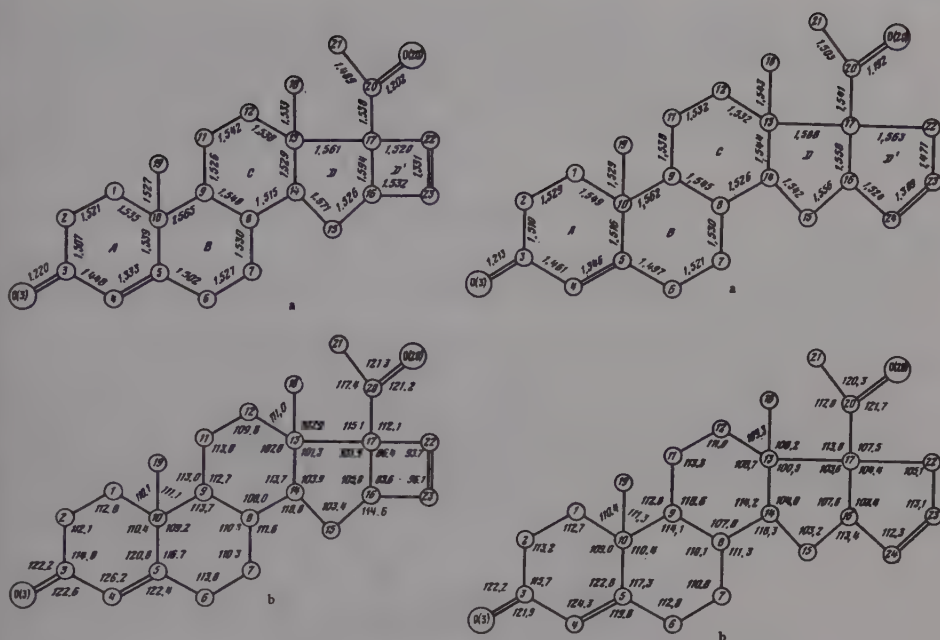


Fig. 1. Interatomic distances (Å) and bond angles (°) in  $C_{23}H_{30}O_2$  (left) and  $C_{24}H_{32}O_2$  (right).

PREGNA-4,16-DIEN-21-OL-3,20-DIONE ACETATE  
 $C_{23}H_{30}O_4$  (I)

PREGNA-1,4-DIEN-17 $\alpha$ ,21-DIOL-3,20-DIONE 21-ACETATE  
 $C_{23}H_{30}O_5$  (II)

Z.I. KHAZHEEVA, V.I. SIMONOV, A.V. KAMERNITSKII, N.S. PAVLOVA-GRISHINA and A.K. SKOROVA, 1982. Kristallografiya, 27, 905-910 [Sov. Phys. Crystallogr., 27, 543-546].

I. Orthorhombic,  $P2_12_12_1$ ,  $a = 7.400$ ,  $b = 12.926$ ,  $c = 21.316$  Å,  $Z = 4$ . Cu radiation,  $R = 0.051$  for 1304 reflexions.

II. Orthorhombic,  $P2_12_12_1$ ,  $a = 7.550$ ,  $b = 11.805$ ,  $c = 23.454$  Å,  $Z = 4$ . Cu radiation,  $R = 0.038$  for 1478 reflexions.

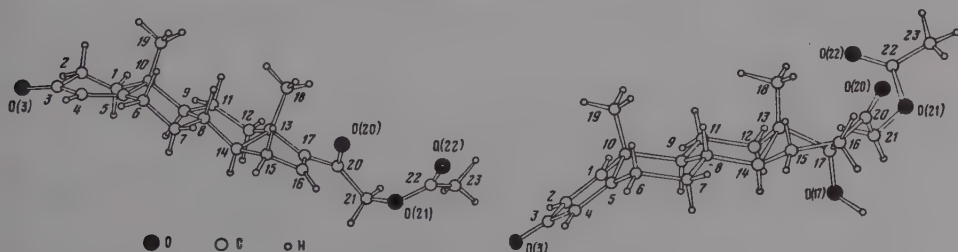
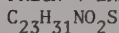


Fig. 1. Projections of  $C_{23}H_{30}O_4$  (left), and of  $C_{23}H_{30}O_5$  (right).

In I (Fig. 1), the A-ring has a  $1\alpha$ -half boat conformation, and the D-ring a  $14\alpha$ -envelope conformation. Bond lengths of note are: C(4)-C(5) 1.342, C(16)-C(17) 1.318 Å. In II (Fig. 1), the A-ring has a boat form and the D-ring is a  $13\beta$ -envelope. Bonds C(1)-C(2), C(4)-C(5) are respectively 1.312 and 1.339 Å. The B and C rings in both molecules have ideal chair conformations. Molecules of II are linked in the crystal by O(17)-H(17)...O(3) hydrogen bonds (O...O 2.748 Å, O-H...O 179.4°) along the b direction.

PREGN-4-ENE-3,20-DIONE-[16,17 $\alpha$ -d]-2'-METHYLTHIAZOLINE



H.A. KARAPETYAN, Yu.T. STRUCHKOV, A.V. KAMERNITZKY and A.M. TURUTA, 1982. Cryst. Struct. Comm., 11, 191-198.

Monoclinic,  $P2_1$ ,  $a = 8.1327$ ,  $b = 7.4455$ ,  $c = 16.980$  Å,  $\beta = 92.46^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.043$  for 1324 reflexions.

Bond lengths and angles in the steroidal skeleton are unexceptional; in the thiazoline ring, bond lengths are  $\text{Csp}^2\text{-S}$  1.792(4),  $\text{Csp}^3\text{-S}$  1.839(4) Å. The molecule is convex on the  $\beta$ -side. Ring A (Fig. 1) has a distorted half-chair conformation, rings B and C are slightly distorted chairs, ring D is intermediate between a  $13\beta$ ,  $14\alpha$  half-chair and a  $14\alpha$  envelope. The thiazoline ring is essentially planar.

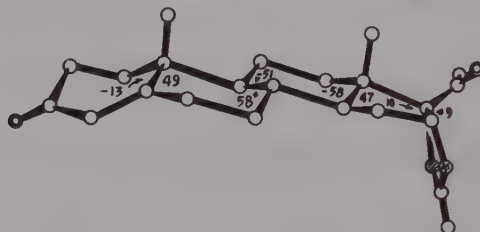


Fig. 1. The  $\text{C}_{23}\text{H}_{31}\text{NO}_2\text{S}$  molecule.

17-OXO-4-ANDROSTENE-3 $\beta$ ,6 $\beta$ -DIOL DIACETATE



W.L. DUAX and P.D. STRONG, 1982. Cryst. Struct. Comm., 11, 1895-1898.

Monoclinic,  $P2_1$ ,  $a = 9.8436$ ,  $b = 13.103$ ,  $c = 8.534$  Å,  $\beta = 103.47^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.058$  for 2243 reflexions.

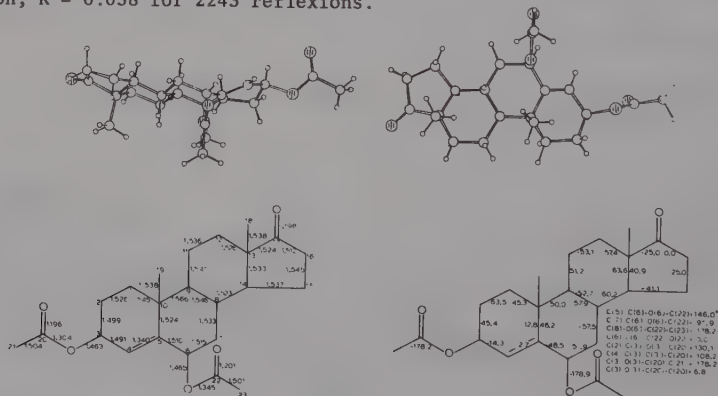
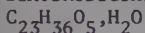


Fig. 1. The  $\text{C}_{23}\text{H}_{32}\text{O}_5$  molecule, with bond lengths and torsion angles.

The analysis shows that the 6 $\beta$ -acetoxy substituent is accommodated without significant alteration of the B-ring chair conformation. Ring A (Fig. 1) is a nearly perfect 1 $\alpha$ ,2 $\beta$ -half-chair, and ring D is a perfect 14 $\alpha$ -envelope.

# DIHYDRODIGOXIGENIN HYDRATE



A. MOSTAD, 1982. Acta Chem. Scand., B36, 635-639.

Orthorhombic,  $P2_12_12_1$ ,  $a = 8.027$ ,  $b = 14.801$ ,  $c = 18.376$  Å,  $Z = 4$ . Mo radiation,  $R = 0.049$  for 1501 reflexions.

The six-membered rings are all in the chair conformation and the D-ring in a 14 $\beta$ -envelope form (Fig. 1). The lactone ring is saturated and hence not planar and the conformation may be described as a C(20)-envelope. The asymmetric centre at C(20) is S in the 3 $\beta$ , 12 $\beta$ , 14 $\beta$  trihydroxy derivative. Each of the three OH groups is involved in two hydrogen bonds, both as donor and acceptor whereas the carbonyl O atom is an acceptor in one hydrogen bond. The O(3)...O(12) bond (2.795 Å) connects molecules in a helix about one of the screw axes, and the O(3)...O(14) (2.670 Å) bond connects molecules in a chain along the  $c$  axis.

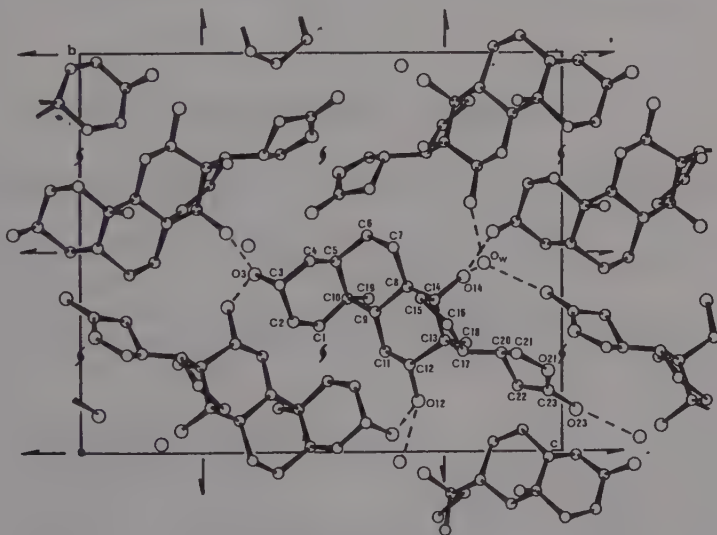


Fig. 1. Packing of dihydrodigoxigenin molecules as seen along  $a$ .

# 16 $\beta$ -ISOPROPYL-16,17-METHANO-4,10 $\beta$ ,13 $\alpha$ -TRIMETHYL-1,2,6,7,10,11,12,13,16,17,3H-DECA-HYDROCYCLOPENTANOPHENANTHRENE-3-ONE



J.P. KUTNEY, P. GRICE, K.O. PASCOE, K. PIOTROWSKA, S. RETTIG, J. SZYKULA and J. TROTTER, 1982. Helv. Chim. Acta, 65, 2345-2354.

Monoclinic,  $P2_1$ ,  $a = 9.022$ ,  $b = 11.477$ ,  $c = 9.636$  Å,  $\beta = 94.540^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.037$  for 1301 reflexions.

The A and C rings (Fig. 1) have distorted C(1) $\beta$  and C(13) $\beta$  sofa-(1,2-diplanar) conformations and ring B has a 1,3-diplanar conformation. The D ring is almost planar (maximum torsion angle  $2.5^\circ$ ). The molecule is substantially bowed, being convex towards the  $a$  face. Bond lengths and angles are generally as expected: mean





3 $\beta$ ,14-DIHYDROXY-5 $\beta$ ,14 $\beta$ -BUFA-20,22-DIENOLIDE (BUFALIN)C<sub>24</sub>H<sub>34</sub>O<sub>4</sub>

D.C. ROHRER, D.S. FULLERTON, E. KITATSUJI, T. NAMBARA and E. YOSHII, 1982. Acta Cryst., B38, 1865-1868.

Orthorhombic, P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, a = 12.381, b = 15.717, c = 10.726 Å, Z = 4. Cu radiation, R = 0.058 for 2195 reflexions.

In the molecule (Fig. 1) the A, B and C rings have the chair conformation while D has a C(14)  $\beta$ -envelope conformation with  $\Delta C_s[C(14)] = 0.2$ . Ring fusions show the cis-trans-cis configuration. The steroid backbone is essentially identical to that in digitoxigenin (1) the cardenolide prototype. The  $\delta$ -lactone ring is planar with the C(13)-C(17)-C(20)-C(22) torsion angle -87.1(3)°. Molecules are linked head-to-tail and thence into helices by hydrogen bonds (O...O 2.907(3) and 3.004(3) Å).

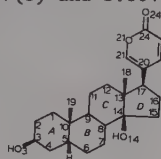
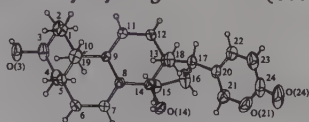


Fig. 1. The bufalin molecule.

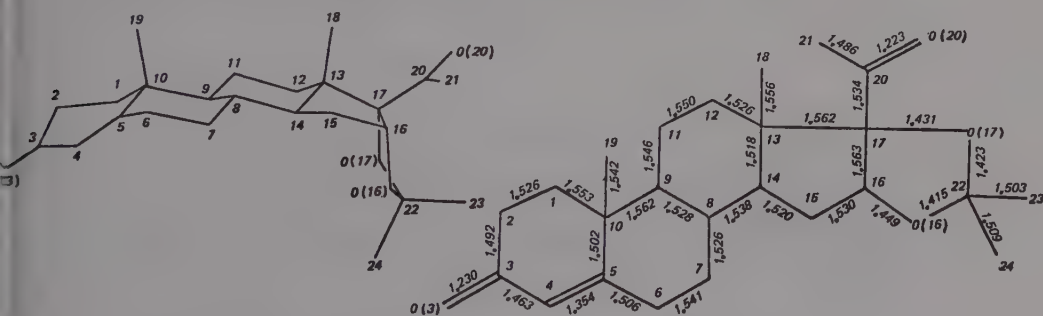
## 1. Structure Reports, 34B, 298.

PREGN-4-ENE-3,20-DIONE-[16 $\alpha$ ,17 $\alpha$ -d]-2',2'-DIMETHYLDIOXALANEC<sub>24</sub>H<sub>34</sub>O<sub>4</sub>

S.V. LINDEMAN, V.E. SHKLOVER, Yu.T. STRUCHKOV, A.M. TURUTA and A.V. KAMERNITZKY, 1982. Cryst. Struct. Comm., 11, 381-386.

Orthorhombic, P2<sub>1</sub>2<sub>1</sub>2, a = 14.651, b = 20.050, c = 7.149 Å, Z = 4. Mo radiation, R = [?] for 1249 reflexions (at -120°C).

Ring A has a distorted 1 $\alpha$ ,2 $\beta$ -half-chair conformation, ring D is a 14 $\alpha$ -envelope and ring E is a C(22) $\beta$ -envelope (Fig. 1).

Fig. 1. The C<sub>24</sub>H<sub>34</sub>O<sub>4</sub> molecule and bond lengths.PREGN-4-ENE-3,20-DIONE-[17 $\alpha$ ,16 $\alpha$ -d]-2',2'-DIMETHYLOXAZOLIDINEC<sub>24</sub>H<sub>35</sub>NO<sub>3</sub>

S.V. LINDEMAN, V.E. SHKLOVER, Yu.T. STRUCHKOV, A.M. TURUTA and A.V. KAMERNITZKY, 1982. Cryst. Struct. Comm., 11, 393-397.

Tetragonal, P4<sub>1</sub>2<sub>1</sub>2, a = 9.016, c = 52.59 Å, Z = 8. Cu radiation, R = 0.040 for 1367 reflexions (at -120°C).

The ring A conformation is intermediate between a  $1\alpha,2\beta$ -half-chair and a  $1\alpha$ -sofa, ring D is intermediate between a  $14\alpha$ -envelope and  $13\beta,14\alpha$ -half-chair, and ring E has a  $C(22)\beta$ -envelope conformation. Bond lengths are shown in Fig. 1.

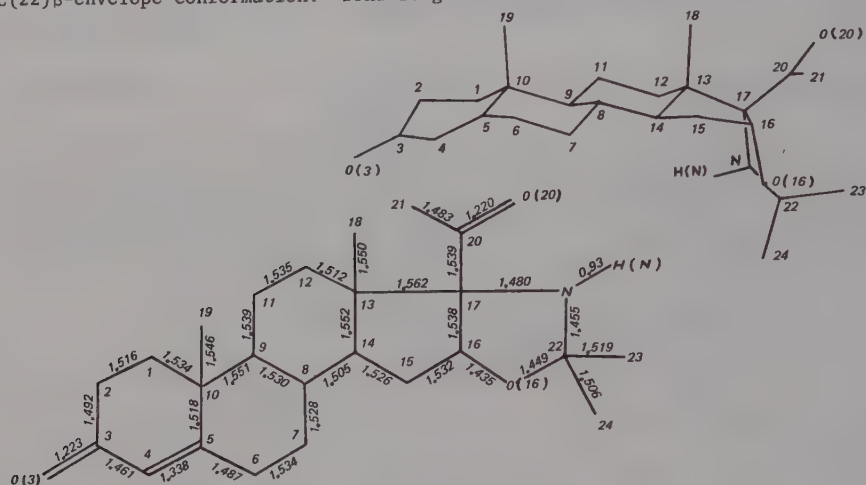


Fig. 1. The  $C_{24}H_{35}NO_3$  molecule and bond lengths.

4,4,14 $\alpha$ -TRIMETHYL-19(10-9 $\beta$ )abeo-5 $\alpha$ ,10 $\alpha$ -PREGNANE-6,11-DIONE  
 $C_{24}H_{38}O_2$

J.R. BULL, D.C. LEVENDIS and A. TUINMAN, 1982. *Tetrahedron*, **38**, 347-352.

Monoclinic,  $P2_1$ ,  $a = 13.988$ ,  $b = 6.411$ ,  $c = 13.933$  Å,  $\beta = 122.37^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.067$  for 1611 reflexions.

The X-ray analysis was used to confirm the conformational results predicted by force field calculations. Details of ring torsion angles are in Fig. 1.

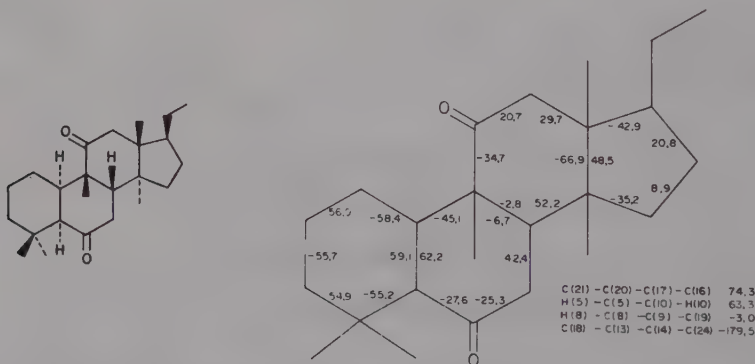
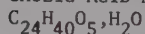


Fig. 1. The  $C_{24}H_{38}O_2$  molecule and torsion angles.

CHOLIC ACID MONOHYDRATE



L. LESSINGER, 1982. *Cryst. Struct. Comm.*, **11**, 1787-1792.

Monoclinic,  $P2_1$ ,  $a = 12.794$ ,  $b = 8.157$ ,  $c = 12.885$  Å,  $\beta = 117.60^\circ$ ,  $D_m = 1.181$ ,  $Z = 2$ . Mo radiation,  $R = 0.069$  for 2925 reflexions.

The crystal structure (Fig. 1) consists of bilayers of cholic acid molecules parallel to the  $bc$  plane, with the water molecules bound up in the complex hydrogen-bonding scheme in the hydrophilic bilayer interior. Each cholic acid molecule is hydrogen-bonded to 4 others, 2 on one side of the bilayer, 2 on the other, and also to 3 water molecules. Each water is at the centre of an approximately tetrahedral arrangement of hydrogen bonds, connecting 3 cholic acid molecules, 1 on one half of the bilayer to 2 on the other. The bilayers are stacked along  $a$ , hydrophobic surfaces in contact. For details of the completely different cholic acid ethanol structure, see (1).

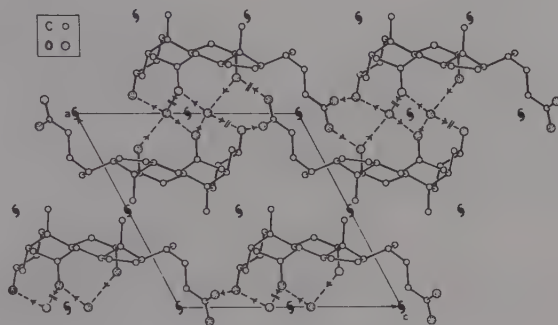


Fig. 1. Packing diagram of cholic acid monohydrate.

1. Structure Reports, 38B, 554.

17a-METHYL-3 $\beta$ -PYRROLIDINYL-17a-AZA-D-HOMO-5 $\alpha$ -ANDROSTANE

$C_{24}H_{42}N_2$

J. HUSAIN, I.J. TICKLE, R.A. PALMER, H. SINGH, T.R. BHARDWAJ and D. PAUL, 1982. Acta Cryst., B38, 130-135.

Triclinic,  $P1$ ,  $a = 7.228$ ,  $b = 10.276$ ,  $c = 15.095$  Å,  $\alpha = 98.377^\circ$ ,  $\beta = 84.690^\circ$ ,  $\gamma = 99.409^\circ$ ,  $D_m = 1.088$ ,  $Z = 2$ . Cu radiation,  $R = 0.054$  for 2271 reflexions.

The two independent molecules in the crystal (Fig. 1) have very similar geometry, the major differences being in the puckering of the pyrrolidine rings and their orientations with respect to the modified steroid skeleton. The methyl carbon atom C(20) on N(17a) is equatorial in both molecules. Intermolecular contacts correspond to van der Waals interactions.

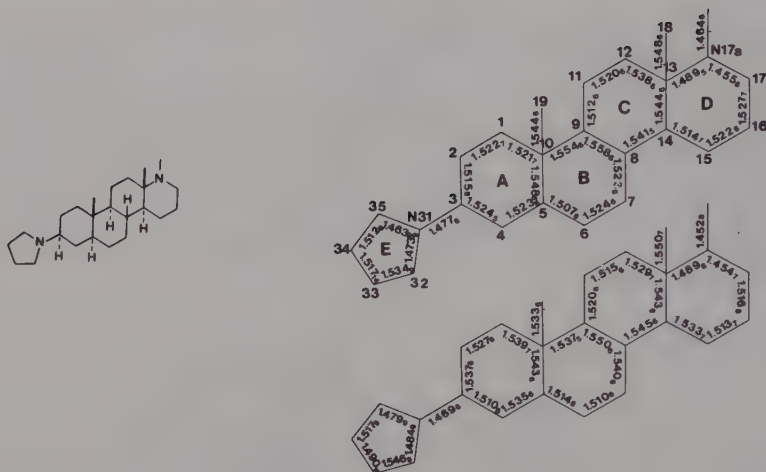


Fig. 1.  $C_{24}H_{42}N_2$ : bond lengths.

20S-3 $\beta$ -20-HYDROXY-12-AZA-C-HOMO-5 $\beta$ -PREGN-12-EN-11-ONE 3,20-DIACETATE ESTER  
 $C_{25}H_{37}NO_5$

R.J. BAKER, L.M. TREFONAS and R.J. MAJESTE, 1982. Cryst. Struct. Comm., **11**, 485-488.

Orthorhombic,  $P2_12_12_1$ ,  $a = 28.289$ ,  $b = 12.1265$ ,  $c = 6.998$  Å,  $D_m = 1.20$ ,  $Z = 4$ . Cu radiation,  $R_w = 0.030$  for 2070 reflexions.

Both six-membered rings (Fig. 1) have chair conformation and are cis-fused. The C(11)-N(12) distance (1.411 Å) is longer than anticipated and there is a localized double bond at N(12)=C(12) (1.267 Å).

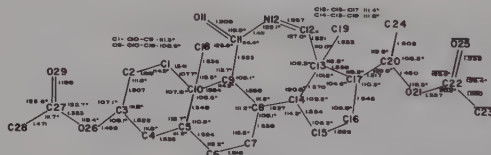


Fig. 1. Bond distances and angles in  $C_{25}H_{37}NO_5$ .

3 $\beta$ ,17 $\alpha$ ,20 $\beta$ -TRIMETHOXY-16 $\beta$ ,23-OXYDO-21,24-DINOR-5 $\alpha$ H-CHOL-6-ONE  
 $C_{25}H_{40}O_5$

V.E. SHKLOVER, S.V. LINDEMAN, Yu.T. STRUCHKOV, A.V. KAMERNITZKY, V.G. LEVY and I.G. RESHETOVA, 1982. Cryst. Struct. Comm., **11**, 1873-1877.

Monoclinic,  $P2_1$ ,  $a = 7.308$ ,  $b = 22.114$ ,  $c = 7.236$  Å,  $\beta = 103.37^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.035$  for 1645 reflexions.

Rings A, B, C and E have somewhat distorted chair conformations (Fig. 1), and ring D is a 1 $\beta$ -envelope.

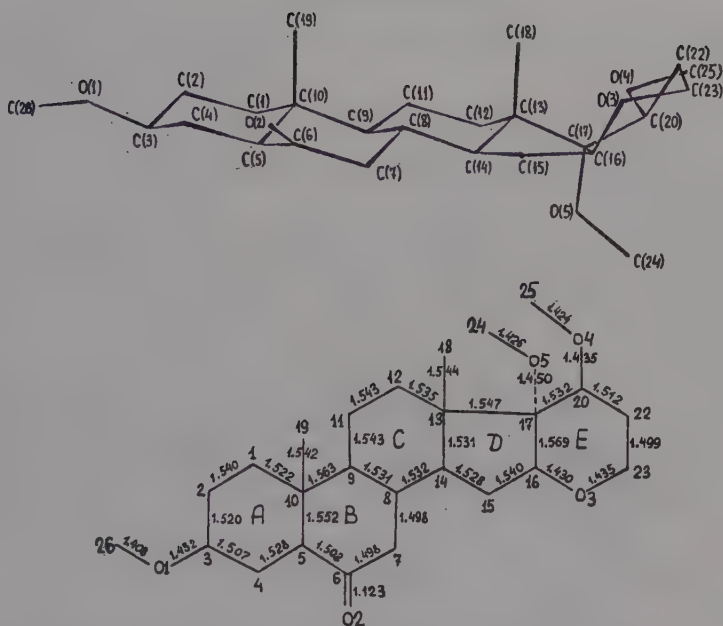


Fig. 1. A view of  $C_{25}H_{40}O_5$  and bond lengths.

21-CORTISOL t-BUTYLACETATE ETHANOL SOLVATE  
 $C_{27}H_{40}O_6 \cdot 0.9(C_4H_{10}O)$

C.-T. LIN, P. PERRIER, G.G. CLAY, P.A. SUTTON and S.R. BYRN, 1982. J. Org. Chem., 47, 2978-2981.

Hexagonal,  $P6_1$ ,  $a = 17.485$ ,  $c = 15.376$  Å,  $Z = 6$ . Cu radiation,  $R = 0.102$  for 1923 reflexions.

The crystal packing of this form has steroid molecules held together by hydrogen bonding arranged in a helix along the  $6_1$  axis. A channel runs through the centre of the helix along the  $6_1$  axis (Fig. 1). The steroid molecules are arranged in helices linked together by  $O29H \cdots O23=C$  hydrogen bonds ( $2.75$  Å) between the carbonyl oxygen of the t-butylacetoxo group and the OH attached to C17.

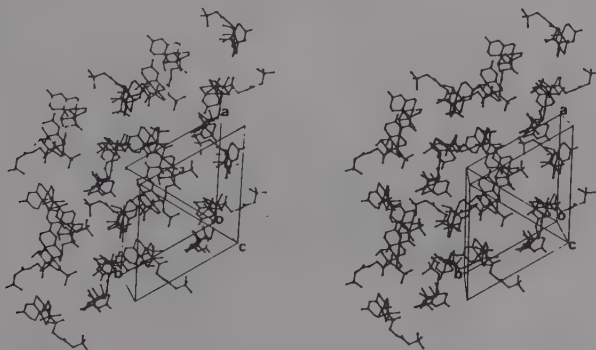


Fig. 1. A stereoview of the crystal packing of cortisol t-butylacetate.



3 $\beta$ -HYDROXY-22-(4-METHYL-1-PYRROLIN-2-YL)-23,24-DINOR-5 $\alpha$ -CHOLANE-4,22-DIONE  
(SOLAMALADINE)

C<sub>27</sub>H<sub>41</sub>NO<sub>3</sub>

A. USUBILLAGA, V. ZABEL and W.H. WATSON, 1982. *Acta Cryst.*, **B38**, 966-969.

Orthorhombic, P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>,  $a = 14.978$ ,  $b = 21.486$ ,  $c = 7.478$  Å,  $Z = 4$ . Cu radiation,  $R = 0.064$  for 1988 reflexions.

In the molecule (Fig. 1) there is little bending of the steroid skeleton. All rings are trans-fused with the six-membered rings in the chair conformation and the five-membered rings in the envelope conformation. Bond lengths and angles are consistent with the structures of other steroids.

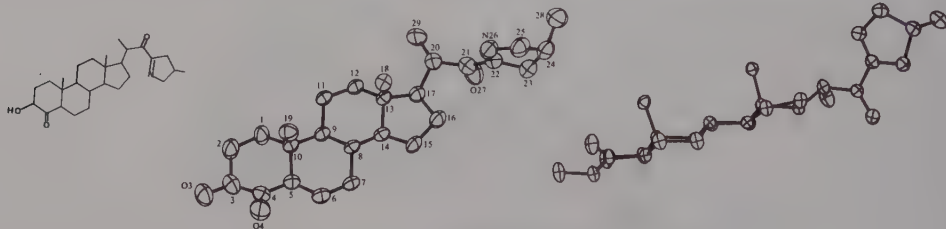


Fig. 1. Perspective views of the solamaladine molecule.

3,6-DIAZA-A,B-BISHOMOCHOLEST-4a-ENO[3,4-d][6,7-d]BISTETRAZOLE MONOHYDRATE

C<sub>27</sub>H<sub>42</sub>N<sub>8</sub>·H<sub>2</sub>O

J. HUSAIN, I.J. TICKLE, R.A. PALMER, H. SINGH and K.K. BHUTANI, 1982. *Acta Cryst.*, **B38**, 2845-2851.

Orthorhombic, P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>,  $a = 9.025$ ,  $b = 12.776$ ,  $c = 50.253$  Å,  $D_m = 1.134$ ,  $Z = 8$ . Cu radiation,  $R = 0.087$  for 6185 reflexions.

In the molecules (Fig. 1) the fusion of tetrazole groups to rings A and B of the steroid has produced strains in the rings resulting in an overall cis conformation. There is disorder in the cholesteryl side chains of both molecules. Solvent water molecules hydrogen-bond with tetrazole groups to form continuous sheets parallel to (001). The two molecules in the asymmetric unit are related by a pseudo twofold axis approximately parallel to [010] and the crystal packing consequently shows pseudo C222<sub>1</sub> symmetry.

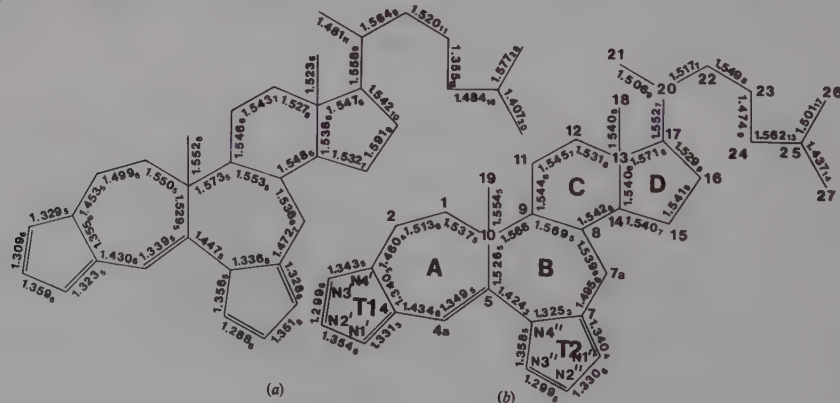
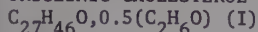


Fig. 1. C<sub>27</sub>H<sub>42</sub>N<sub>8</sub>·H<sub>2</sub>O: bond lengths in molecules A and B of the asymmetric unit.

## TRICLINIC CHOLESTEROL HEMIETHANOLATE



## MONOCLINIC CHOLESTEROL HEMIETHANOLATE



H.S. SHIEH, L.G. HOARD and C.E. NORDMAN, 1982. *Acta Cryst.*, **B38**, 2411-2419.

I. Triclinic,  $P1$ ,  $a = 12.787$ ,  $b = 35.310$ ,  $c = 12.225 \text{ \AA}$ ,  $\alpha = 97.80^\circ$ ,  $\beta = 100.40^\circ$ ,  $\gamma = 99.06^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.095$  for 7291 reflexions.

II. Monoclinic,  $P2_1$ ,  $a = 12.775$ ,  $b = 68.668$ ,  $c = 12.213 \text{ \AA}$ ,  $\beta = 100.43^\circ$ ,  $Z = 16$ . Cu radiation,  $R = 0.106$  for 6093 reflexions.

Both structures are characteristic bilayer structures with alternating hydrophobic and hydrophilic layers; in the latter the cholesterol and ethanol molecules are joined into hydrogen-bonded chains. Both I and II exhibit translational pseudosymmetry within their asymmetric units, whereby one pair of cholesterol molecules is related to another pair by an approximate  $a/2$  translation, while the third and fourth pairs are related by an approximate  $c/2$  translation. This pseudosymmetry is similar to, but less closely obeyed than, the pseudosymmetry in cholesterol monohydrate (1). Average bond lengths and angles are shown in Fig. 1.

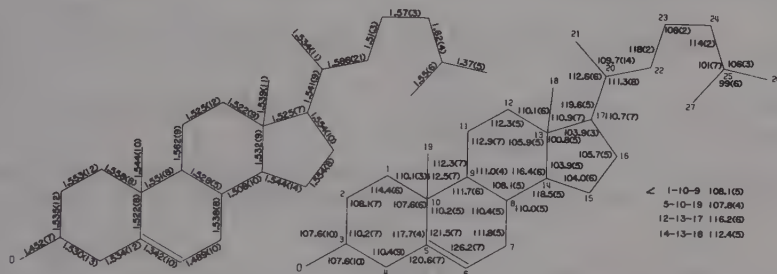
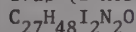


Fig. 1.  $\text{C}_{27}\text{H}_{46}\text{O}_2 \cdot 0.5(\text{C}_2\text{H}_6\text{O})$ : bond lengths and angles, averaged over the 16 independent cholesterol molecules in the triclinic and monoclinic structures.

1. Structure Reports, **45B**, 576.

17 $\alpha$ B-(2-HYDROXYETHYL)-3 $\beta$ -PYRROLIDINO-17 $\alpha$ -AZA-d-HOMO-5-ANDROSTENE DIMETHIODIDE



A.I. EL-SHORA, R.A. PALMER, H. SINGH, T.R. BHARDWAJ and D. PAUL, 1982. *J. Cryst. Spectrosc. Res.*, **12**, 255-270.

Orthorhombic,  $P2_12_12_1$ ,  $a = 14.671$ ,  $b = 17.594$ ,  $c = 10.908 \text{ \AA}$ ,  $Z = 4$ . Mo radiation,  $R = 0.044$  for 3041 reflexions.

Rings A, C, and D-homo (Fig. 1) have chair conformations, ring B is a half-chair and the pyrrolidine ring E has an envelope conformation. The  $\text{N}^+ \dots \text{N}^+$  distance of  $10.33 \text{ \AA}$  is within the usual range associated with potent neuromuscular blocking. The hydroxyethyl side group appended to  $\text{N}^+(17a)$  forms part of an acetylcholine-like moiety which is observed to adopt the  $t, -g$  conformation commonly found in acetylcholine. Potential-energy calculations for the free molecule indicate that the side chain may adopt a variety of conformations in the range  $t, (+g \text{ to } -g)$ .



The A and C rings adopt chair conformations, ring B is a 5 $\alpha$ ,6 $\beta$ -half-chair and ring D approximates a 13 $\beta$ -envelope. Bond distances are in Fig. 1. There are four intermolecular hydrogen bonds giving rise to bilayers.

3 $\alpha$ ,5-EPOXY-6 $\beta$ -IODO-3 $\beta$ -METHYL-A-HOMO-4-OXA-5 $\alpha$ -CHOLESTANE

C<sub>28</sub>H<sub>47</sub>IO<sub>2</sub>

A. FURUSAKI, C. KATAYAMA and H. SUGINOME, 1982. Bull. Chem. Soc. Jpn., **55**, 3041-3042.

Orthorhombic, P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, a = 17.44, b = 21.27, c = 7.26 Å, Z = 4. Mo radiation, R = 0.064 for 1786 reflexions.

Rings A, B and C have chair conformations, ring D has half-chair and ring E has an envelope conformation (Fig. 1). Ring A is deformed by the bridging CH<sub>2</sub>-O moiety. The side-chain at C17 has an extended zig-zag conformation.

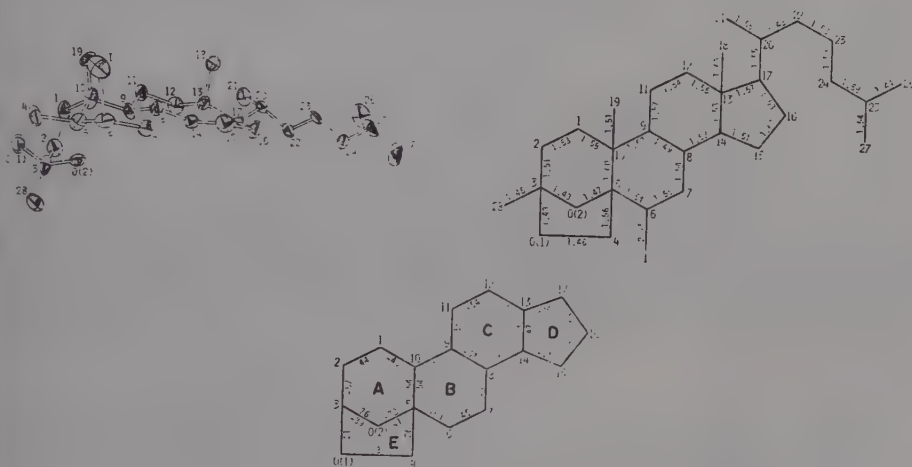


Fig. 1. A view of C<sub>28</sub>H<sub>47</sub>IO<sub>2</sub> with bond distances and torsion angles.

HUMISTRATIN HEMIHYDRATE

C<sub>29</sub>H<sub>38</sub>O<sub>9</sub>·0.5H<sub>2</sub>O

S. NISHIO, M.S. BLUM, J.V. SILVERTON and R.J. HIGHET, 1982. J. Org. Chem., **47**, 2154-2157.

Orthorhombic, B22<sub>1</sub>2, a = 12.216, b = 19.216, c = 22.970 Å, D<sub>m</sub> = 1.31, Z = 8. Cu radiation, R = 0.034 for 2427 reflexions.

The analysis establishes the structure shown in Fig. 1. The six-membered rings, apart from ring B, all adopt fairly standard chair conformations and ring B has the usual cyclohexene monoplanar conformation. The D ring of the steroid moiety has a 14-envelope conformation and the lactone ring is almost flat.

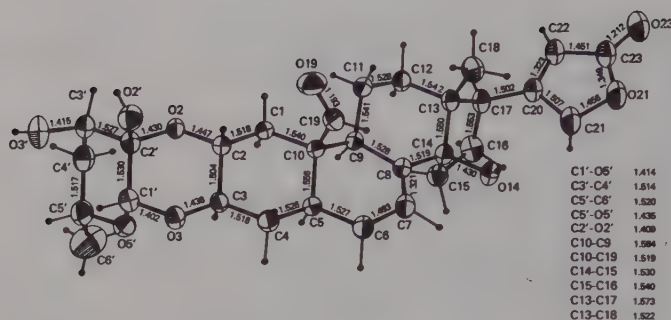


Fig. 1. Bond lengths in humistratin.

9 $\alpha$ -FLUORO-11 $\beta$ -HYDROXY-16 $\beta$ -PROPIONYLOXY-17-(2-ETHYL-2-ETHOXY-1,3-DIOXAN-5-ONE-6-YL)-PREGNA-1,4-DIEN-3-ONE

$C_{29}H_{39}FO_8$

G. BANDOLI and M. NICOLINI, 1982. *J. Cryst. Spectrosc. Res.*, **11**, 321-329.

Orthorhombic,  $P2_12_12_1$ ,  $a = 15.992$ ,  $b = 15.693$ ,  $c = 11.205$  Å,  $D_m = 1.25$ ,  $Z = 4$ . Mo radiation,  $R = 0.085$  for 1216 reflexions.

Strain in the almost planar ring A (Fig. 1) is indicated by the endocyclic torsion angles, rings B and C, which are trans fused and chair-shaped, while ring D is intermediate between a 13 $\beta$ -envelope and a 13 $\beta$ ,14 $\alpha$ -half-chair conformation. Ring E, a 2,5,6-trisubstituted 1,3-dioxane-like unit, adopts a highly distorted boat conformation. The bending of the skeleton is toward its  $\alpha$  side. The A-ring carbonyl substituent accepts one hydrogen bond involving a symmetry-related 11 $\beta$ -ol function (O(1)...O(2) 2.81 Å).

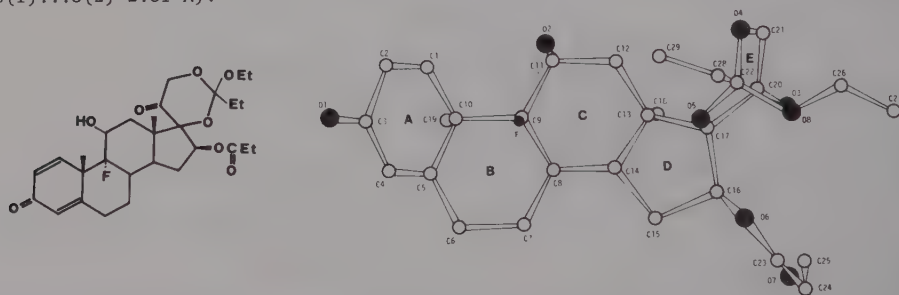


Fig. 1. The  $C_{29}H_{39}FO_8$  molecule.

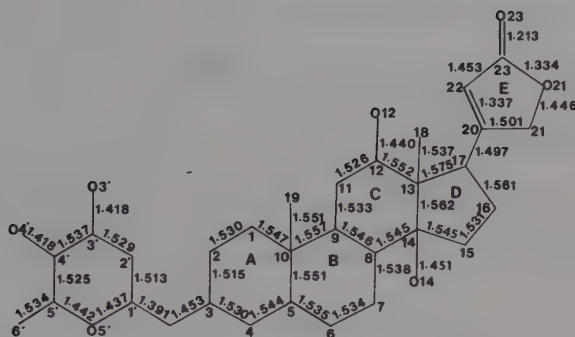
DIGOXIGENIN MONODIGITOXOSIDE HYDRATE

$C_{29}H_{44}O_8 \cdot H_2O$

K. GO and G. KARTHA, 1982. *Cryst. Struct. Comm.*, **11**, 279-284.

Monoclinic,  $P2_1$ ,  $a = 7.341$ ,  $b = 13.811$ ,  $c = 13.811$  Å,  $\beta = 104.8^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.060$  for 2864 reflexions.

Rings A and D are cis with respect to the fused B and C rings (Fig. 1). Five-membered ring D is a 13 $\beta$ -envelope. The orientation of the lactone ring is defined by the C(13)-C(17)-C(20)-C(22) torsion angle,  $-95.7^\circ$ . The water of hydration is hydrogen bonded to O(3') of the monosaccharide (O...O 2.891 Å).





5 $\alpha$ -LANOST-24-ENE-3 $\beta$ ,11 $\beta$ -DIOL 3-CHLOROACETATE  
 $C_{32}H_{53}ClO_3$

R.B. BOAR, D.B. COPSEY, P.F. LINDLEY and R.C. HORSBURGH, 1982. J. Chem. Research, S, 322-323; M, 3307-3341.

Monoclinic,  $P2_1$ ,  $a = 18.340$ ,  $b = 6.086$ ,  $c = 13.933$  Å,  $\beta = 96.35^\circ$ ,  $Z = 2$ .  $R = 0.08$ .

Rings A, B, and C of the molecule (Fig. 1) adopt a chair conformation and ring D an almost perfect 13 $\beta$ ,14 $\alpha$ -half chair conformation. The nucleus exhibits a slight twist along the backbone with a torsion angle of  $5^\circ$ . Side-chain atoms did not refine satisfactorily. Otherwise bond lengths and angles are normal with e.s.d.'s of 0.02 Å and 0.9-1.4°.

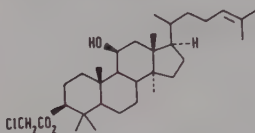


Fig. 1.  $C_{32}H_{53}ClO_3$ : the molecular skeleton.

3 $\beta$ -ACETOXY-15 $\alpha$ -(*o*-NITROBENZOYL)OXY-5 $\beta$ ,14 $\beta$ -BUFA-8,20,22-TRIENOLIDE  
 $C_{33}H_{37}NO_8$

L.R. NASSIMBENI, M.L. NIVEN, G.R. PETTIT, Y. KAMANO, M. INOVE and J.J. EINCK, 1982. Acta Cryst., B38, 2163-2166.

Orthorhombic,  $P2_12_12_1$ ,  $a = 31.53$ ,  $b = 11.533$ ,  $c = 8.221$  Å,  $D_m = 1.27$ ,  $Z = 4$ . Mo radiation,  $R = 0.095$  for 1383 reflexions.

In the molecule (Fig. 1) there is cis fusion of the A/B and C/D rings, while the B/C rings are fused by a double bond, C(8)-C(9) 1.38(1) Å. Bond lengths and angles are in good agreement with accepted values. No intermolecular contacts are less than 3.5 Å.

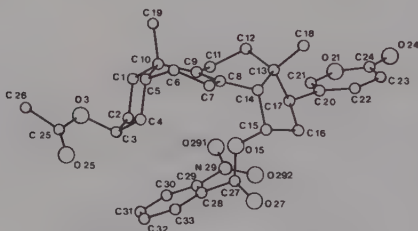


Fig. 1.  $C_{33}H_{37}NO_8$ : molecular structure.

CHOLESTERYL BENZOATE  
 $C_{34}H_{50}O_2$

A.P. POLISHCHUK, V.I. KULISHOV and A.A. MOISEENKO, 1982. Cryst. Struct. Comm., 11, 2069-2073.

Tetragonal,  $P4_12_12$ ,  $a = 10.520$ ,  $c = 54.42$  Å,  $Z = 8$ . Cu radiation,  $R = 0.106$  for 2553 reflexions.

The conformation of the steroid nucleus (Fig. 1) is typical for a  $\Delta^5$ -steroid. The molecules are packed to form layers which are parallel to the (001) crystal planes. The crystal structure is very similar to the orthorhombic modification of cholesteryl-*m*-bromobenzoate (1). The long molecular axis running through the C25 and C32 atoms makes an angle  $57^\circ$  with the *c*-axis along which the molecules are repeated

by the  $4_1$  axis. Such a packing is important because a similar arrangement may occur in the chiral smectic C phase.

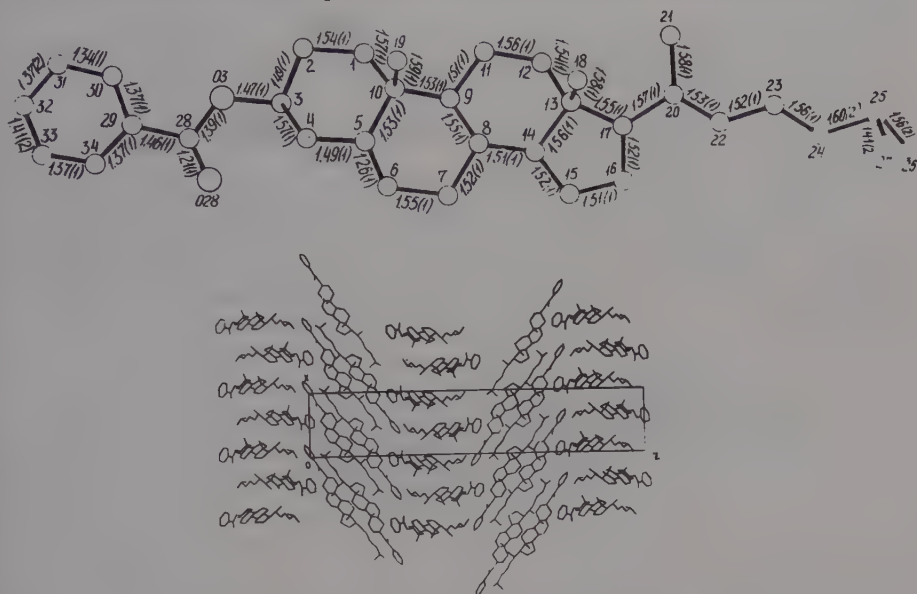
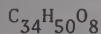


Fig. 1. Molecular dimensions and a packing diagram for  $C_{34}H_{50}O_2$ .

1. Structure Reports, 48B, 662.

#### NOR-CYASTERONE DIACETONIDE



C. MIRAVITLES, X. SOLANS, G. GERMAIN and J.P. DECLERCQ, 1982. Cryst. Struct. Comm., 11, 1683-1688.

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.42$ ,  $b = 11.25$ ,  $c = 40.39$  Å,  $Z = 4$ . Mo radiation,  $R = 0.079$  for 1124 reflexions.

The analysis confirms a structure established by chemical and spectral data. Rings A and B are in a distorted half-chair conformation, ring C is a distorted sofa and ring D is in a half-chair conformation (Fig. 1).

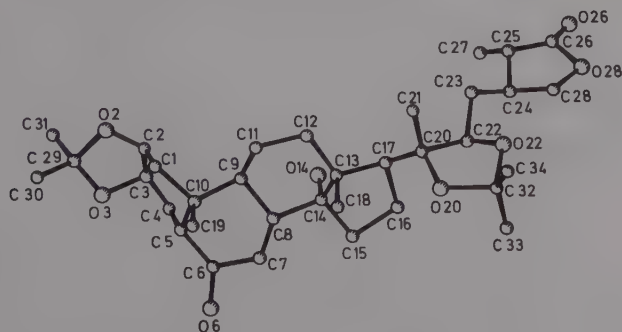
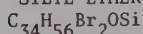


Fig. 1. A view of the  $C_{34}H_{50}O_8$  molecule.

22 $\alpha$ ,23 $\alpha$ -DIBROMO-18-NOR-12-METHYL-5 $\alpha$ ,17 $\beta$ -ERGOST-8,11,13(14)-TRIEN-3 $\beta$ -t-BUTYLDIMETHYLSILYL ETHER



G. FERGUSON, P.Y. SIEW, W.B. WHALLEY and C.L. YEATES, 1982. Cryst. Struct. Comm., 11, 775-780.

Monoclinic,  $P2_1$ ,  $a = 11.083$ ,  $b = 11.605$ ,  $c = 14.185 \text{ \AA}$ ,  $\beta = 101.22^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.099$  for 1526 reflexions.

The crystal structure contains discrete molecules separated by van der Waals distances. The disorder of the silyl ether moiety and crystal decomposition during data collection limited the accuracy obtained by the analysis. The molecular dimensions are unexceptional and the structure very closely resembles that found for the corresponding 3 $\beta$ -acetate derivative (1) even to the conformation of the side chain at C(17).

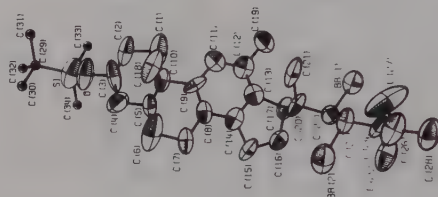
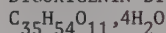


Fig. 1. A view of  $\text{C}_{34}\text{H}_{56}\text{Br}_2\text{OSi}$ .

1. Structure Reports, 29, 751.

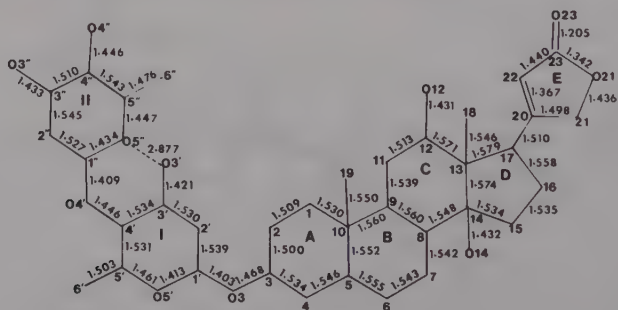
DIGOXIGENIN BISDIGITOXOSIDE TETRAHYDRATE



K. GO and G. KARTHA, 1982. Struct. Comm., 11, 285-290.

Orthorhombic,  $P2_12_12_1$ ,  $a = 35.715$ ,  $b = 14.422$ ,  $c = 7.526 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.100$  for 2551 reflexions.

The A and D rings are cis with respect to the fused B and C rings (Fig. 1). There is an intramolecular hydrogen bond between the hydroxyl at C(3') of the first sugar and the ring oxygen of the second sugar ( $2.877(5) \text{ \AA}$ ). The D-ring conformation is between a  $15\alpha$ -envelope and a  $14\beta,15\alpha$ -distorted half-chair. The lactone ring orientation is defined by the torsion angle C(13)-C(17)-C(20)-C(22) of  $-94.2^\circ$ . The water molecules, two of which are disordered, are involved in a three-dimensional hydrogen-bonded network.



2 $\alpha$ ,3 $\alpha$ ,22S,23S-BIS(ISOPROPYLIDENEDIOXY)-24S-ETHYL-5 $\alpha$ -CHOLESTAN-6-ONE  
 $C_{35}H_{58}O_5$

K. MORI, M. SAKAKIBARA, Y. ICHIKAWA, H. UEDA, K. OKADA, T. UMEMURA, G. YABUTA, S.K.M. KONDO, M. MINOBE and A. SOGABE, 1982. Tetrahedron, 38, 2099-2109.

Monoclinic,  $P2_1$ ,  $a = 18.228$ ,  $b = 7.598$ ,  $c = 12.577$  Å,  $\beta = 105.2^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.058$  for 1891- reflexions:

The analysis clarifies the stereochemistry of the side-chain portion of the molecule as shown in Fig. 1.

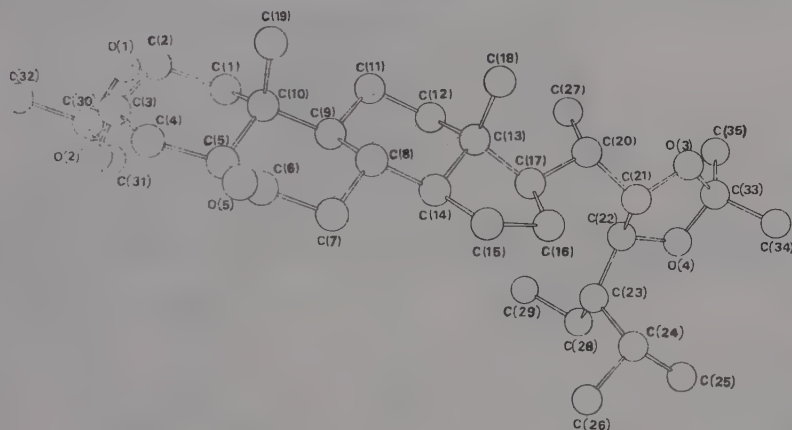


Fig. 1. A view of  $C_{35}H_{58}O_5$ .

CHOLEST-5-en-3-ol-p-CHLOROCINNAMATE  
 $C_{36}H_{51}ClO_2$

A.P. POLISHCHUK, V.I. KULISHOV, R.M. CHERKASHINA, M.Yu. ANTIPIN and Yu.T. STRUCHKOV, 1982. Cryst. Struct. Comm., 11, 1737-1742.

Monoclinic,  $P2_1$ ,  $a = 11.80$ ,  $b = 9.81$ ,  $c = 13.92$  Å,  $\beta = 101.5^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.124$  for 1327 reflexions (at  $-120^\circ C$ ).

The A and C rings (Fig. 1) have chair conformation, ring D is a C13 envelope. In the B ring C(9),C(10),C(5),C(6) are coplanar within 0.01 Å, with C(7) 0.39, and C(8) 0.89 Å from this plane.

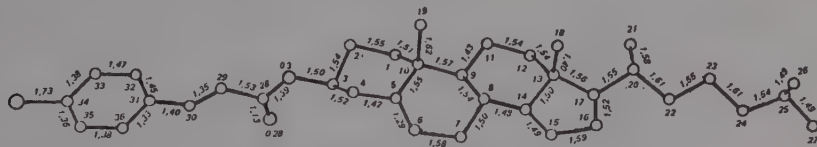


Fig. 1. A view of  $C_{36}H_{51}ClO_2$  and bond lengths ( $\sigma$  0.02-0.04 Å).

CHOLESTERYL PALMITOLEATE  
 $C_{43}H_{74}O_2$

P. SAWZIK and B.M. CRAVEN, 1982. Acta Cryst., B38, 1777-1781.

Monoclinic,  $P2_1$ ,  $a = 12.873$ ,  $b = 9.173$ ,  $c = 35.424$  Å,  $\beta = 93.47^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.122$  for 2383 reflexions (at 295 K).

Molecules are packed in an antiparallel array forming monolayers parallel to (001) (35.4 Å thick). Central regions of the monolayers show efficient molecular packing with adjacent molecules having their tetracyclic ring systems almost perpendicular to each other (Fig. 1). The layer interface regions are more loosely packed and have conformational disordering in the terminal iso-propyl group in the (A) tail and in a five-atom segment of the (B) ester chain around the cis double bond. There is a 47° difference in rotation about the ester bond in the two molecules. Molecule A has a kinked, nearly extended ester chain while the disordered chains in B have two different bent conformations. Molecular geometry is normal with molecular lengths: A, 34.15(8) and B, 33.33(9) Å.

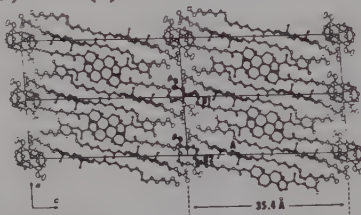
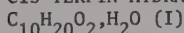
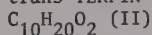


Fig. 1. Crystal structure of cholesteryl palmitoleate down the b-axis. The disordered conformers (B1) and (B2) are shown separately (shaded) and together (unshaded).

#### cis-TERPIN HYDRATE



#### trans-TERPIN



T. SUGA, T. HIRATA and T. AOKI, 1982. Bull. Chem. Soc. Jpn., 55, 914-917.

I. Orthorhombic, Fdd2,  $a = 10.930$ ,  $b = 18.425$ ,  $c = 22.791$  Å,  $D_m = 1.08$ ,  $Z = 16$ . Mo radiation,  $R = 0.044$  for 1091 reflexions.

II. Monoclinic, C2/c,  $a = 17.167$ ,  $b = 6.233$ ,  $c = 19.325$  Å,  $\beta = 98.910^\circ$ ,  $D_m = 1.16$ ,  $Z = 8$ . Mo radiation,  $R = 0.049$  for 1261 reflexions.

The analyses shows that in I the OH group at C1 is axial and the C4 substituent equatorial, whereas in II the OH at C1 and the C4 substituent are equatorial (Fig. 1).

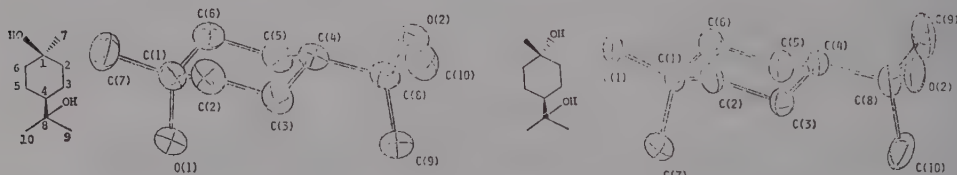
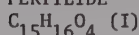
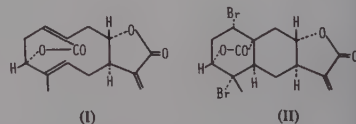
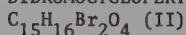


Fig. 1. The cis- (1) and trans- (2) terpin molecules.

#### PERTILIDE



#### DIBROMOCYCLOPERTILIDE



S. NAGUMO, K.-I. KAWAI and Y. IITAKA, 1982. Acta Cryst., B38, 1660-1662.

I. Orthorhombic,  $P2_12_12_1$ ,  $a = 8.278$ ,  $b = 25.244$ ,  $c = 6.179$  Å,  $Z = 4$ . Cu radiation,  $R = 0.083$  for 1161 reflexions.

II. Orthorhombic,  $P2_12_12_1$ ,  $a = 14.655$ ,  $b = 16.089$ ,  $c = 6.558$  Å,  $Z = 4$ . Cu radiation,  $R = 0.078$  for 1507 reflexions.

In (I) the trans,trans-cyclodeca-1,5-diene ring has C(14) and C(15) anti with respect to the ring plane (Fig. 1). The two double bonds of the ten-membered ring are in close contact (centre-to-centre distance, 2.94 Å). The atoms C(1), C(4), C(5), C(10) are planar within 0.08 Å. In (II) the three, condensed, six-membered rings have slightly deformed boat conformations, the central six-membered ring is a chair and the  $\gamma$ -lactone has a much less planar envelope form than in (I).

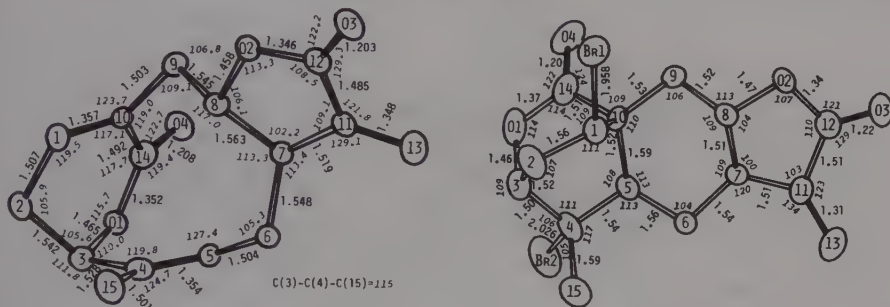


Fig. 1. The molecular geometry of C<sub>15</sub>H<sub>16</sub>O<sub>4</sub> (I) (left) and of C<sub>15</sub>H<sub>16</sub>Br<sub>2</sub>O<sub>4</sub> (II) (right).

# OBTUSALLENE C<sub>15</sub>H<sub>17</sub>Br<sub>2</sub>ClO<sub>2</sub>

P.J. COX and R.A. HOWIE, 1982. *Acta Cryst.*, B38, 1386-1387.

Orthorhombic,  $P2_12_12_1$ ,  $a = 5.545$ ,  $b = 10.082$ ,  $c = 28.589$  Å,  $Z = 4$ . Mo radiation,  $R = 0.082$  for 1218 reflexions.

The molecule (Fig. 1) may be described as a halogenated bicyclic ether with an allenic side chain with C(14)-C(15) 1.32(3), C(15)-C(16) 1.35(3) Å and C(14)-C(15)-C(16) 179.5°. The dihydropyran ring adopts a half-chair conformation. The C(1)...O(5) transannular separation (2.84 Å) is distinctly short. The only short intermolecular separation is Br(2)...Cl' 3.539 Å.

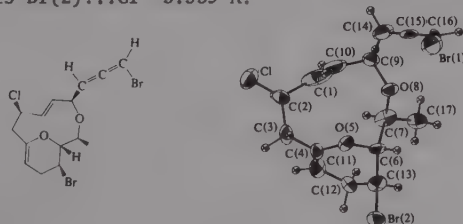


Fig. 1. C<sub>15</sub>H<sub>17</sub>Br<sub>2</sub>ClO<sub>2</sub>: a perspective view of the molecule.

# FRUTESCIN C<sub>15</sub>H<sub>18</sub>O<sub>3</sub>

W. HERZ, J.S. PRASAD and J.F. BLOUNT, 1982. *J. Org. Chem.*, 47, 2206-2208.



Orthorhombic,  $P2_12_12_1$ ,  $a = 7.684$ ,  $b = 10.506$ ,  $c = 16.092$  Å,  $Z = 4$ . Cu radiation,  $R = 0.033$  for 955 reflexions.

The  $\gamma$ -lactone ring of 2 is quite puckered, the sum of the internal torsion angles being  $98^\circ$ , and is described as a twist envelope with C-7 as the flap and C-8 slightly depressed from the C-8, O-1, C-12, C-11 plane. The conformation of the ten-membered ring to which the lactone ring is attached pseudodiequatorially is somewhat less strained than and quite different from that exhibited by comparable C-6 lactonized melampolides. The C-4 methyl and the C-10 aldehyde group are syn (and  $\beta$  to the plane of the ring) but project away from each other as the result of a small movement of C-3 and C-6 out of the nodal plane of the C-4, C-5 double bond which produces a torsion angle of  $168^\circ$ . The conformation in solution does not appear to depart significantly from that observed in the crystal.

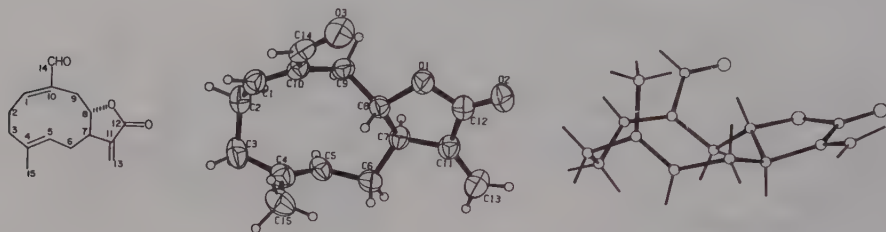
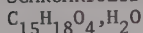


Fig. 1. Frutescin.

#### SCHKUHLRIOLID MONOHYDRATE



U. RYCHLEWSKA, 1982. J. Chem. Soc. Perkin II, 1641-1644.

Orthorhombic,  $P2_12_12_1$ ,  $a = 6.968$ ,  $b = 9.603$ ,  $c = 22.478$  Å,  $D_m = 1.23$ ,  $Z = 4$ . Cu radiation,  $R = 0.053$  for 1136 reflexions.

The structure and absolute configuration of the germacranolide derivative (Fig. 1) from "*Schkuhria schkuhrioides*" have been established. The unusual cis-fusion of the lactone ring causes little change in the conformation of the ten membered ring compared with that of related trans-fused compounds. This ring has a distorted chair-boat conformation with the substituents at C(4) and C(10) anti. Minor changes due to the cis fusion are the smaller C(6)-C(7)-C(8)-C(9) torsion angle ( $-38^\circ$ ) and the expanded C(7)-C(8)-C(9) bond angle ( $124.8^\circ$ ). The  $\gamma$ -lactone is a half chair with a pseudo-diad passing through C(12) and the mid point of C(7)-C(8), and with bond angles C(7)-C(11)-C(12)  $106.5^\circ$ , C(11)-C(12)-O(1)  $109.3^\circ$ , C(12)-O(1)-C(8)  $108.6^\circ$ , O(1)-C(8)-C(7)  $103.6^\circ$  and C(8)-C(7)-C(11)  $98.8^\circ$ . There is extensive hydrogen bonding in the crystal, involving the lactone carbonyl and the solvent water.

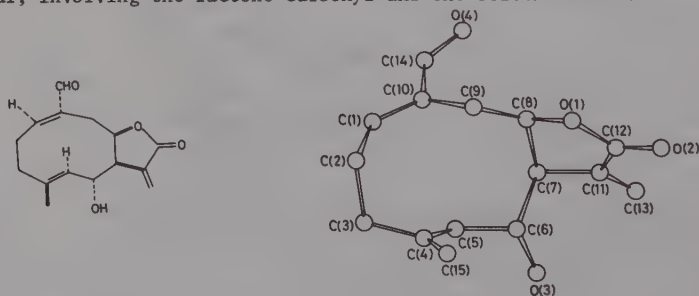


Fig. 1. Molecular skeleton and structure of  $C_{15}H_{18}O_4 \cdot H_2O$ .

## BERMUDENYOL

 $C_{15}H_{19}BrCl_2O_2$ 

J.H. CARDELLINA, S.B. HORSLEY, J. CLARDY, S.R. LEFTOW and J. MEINWALD, 1982.  
 Canad. J. Chem., 60, 2675-2677.

Monoclinic,  $P2_1$ ,  $a = 10.52$ ,  $b = 5.57$ ,  $c = 15.80$  Å,  $\beta = 108.97^\circ$ ,  $Z = 2$ . Mo radiation,  
 $R = 0.104$  for 1306 reflexions.

The analysis confirms the structure assigned to bermudenylol and establishes the stereochemistry to be as shown in Fig. 1.

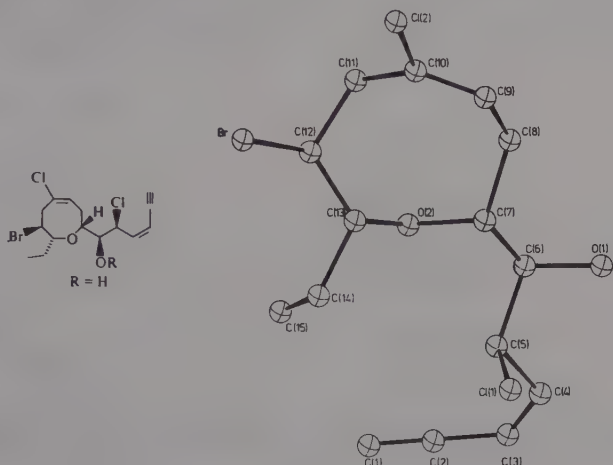


Fig. 1. The structure of bermudenylol.

## trans-PINNATIFIDENYNE

 $C_{15}H_{20}BrClO$ 

A.G. GONZÁLEZ, J.D. MARTÍN, V.S. MARTÍN, M. NORTE, R. PÉREZ, J.Z. RUANO, S.A.  
 DREXLER and J. CLARDY, 1982. Tetrahedron, 38, 1009-1014.

Triclinic,  $C1$ ,  $a = 5.454$ ,  $b = 8.882$ ,  $c = 10.072$  Å,  $\alpha = 105.97^\circ$ ,  $\beta = 68.86^\circ$ ,  $\gamma = 75.64^\circ$ ,  
 $Z = 1$ .  $R = 0.084$  for 1010 reflexions.

The analysis establishes the structure of this metabolite from red seaweed as shown in Fig. 1.

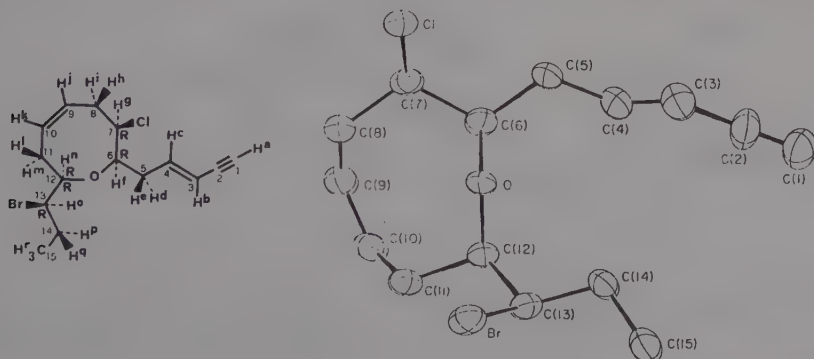


Fig. 1. trans-Pinnatifidenyne.

## ARISTOLACTONE



G. FERGUSON, P. GALATSIS, G.L. LANGE and B.L. RUHL, 1982. J. Chem. Research, S, 304-305; M, 3261-3282.

Orthorhombic,  $P2_12_12_1$ ,  $a = 6.543$ ,  $b = 13.986$ ,  $c = 14.735$  Å,  $Z = 4$ . Mo radiation,  $R = 0.035$  for 785 reflexions.

The X-ray analysis establishes the relative stereochemistry at C(6) and C(7) (Fig. 1) to be in the cis-configuration. The molecular dimensions have acceptable values with  $C(sp^3)-C(sp^3)$  1.545(5)-1.555(5),  $C(sp^3)-C(sp^2)$  1.454(6)-1.507(6),  $C(sp^2)-C(sp^2)$  1.446(6),  $C(sp^2)=C(sp^2)$  1.316(5)-1.332(6),  $C(sp^3)-O$  1.446(5),  $C(sp^2)-O$  1.359(5) and  $C=O$  1.213(5) Å. The conformation of the ten-membered ring is defined by the torsion angles.

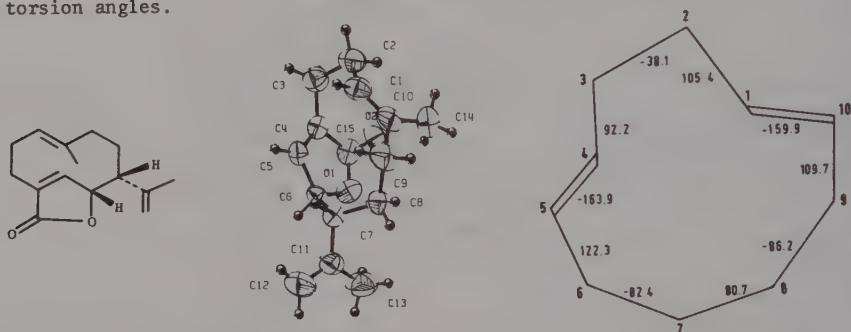


Fig. 1.  $C_{15}H_{20}O_2$ : the molecular skeleton, structure and torsion angles.

4,4a $\beta$ ,5,6,7,8-HEXAHYDRO-4 $\beta$ ,5 $\alpha$ ,7 $\beta$ -TRIMETHYL-3-ETHYLIDENE-7 $\alpha$ -HYDROXYNAPHTHALENE-2-ONE  
 $C_{15}H_{22}O_2$

E.O. SCHLEMPER, H. KOOSHKABADI, B. NASSIM and P. CRABBE, 1982. Cryst. Struct. Comm., 11, 65-70.

Monoclinic,  $P2_1/n$ ,  $a = 10.775$ ,  $b = 10.801$ ,  $c = 12.221$  Å,  $\beta = 108.02^\circ$ ,  $D_m = 1.09$ ,  $Z = 4$ . Mo radiation,  $R = 0.042$  for 1364 reflexions.

The saturated six-membered ring (Fig. 1) is in normal chair conformation while the other ring is nearly planar. Molecules are linked by O-H...O hydrogen bonds (O...O 2.853(3) Å).

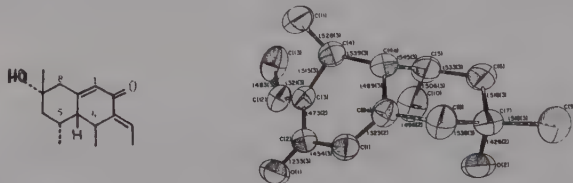


Fig. 1. The  $C_{15}H_{22}O_2$  molecule.

## IVAXILLIN



W. HERZ, J.S. PRASAD and J.F. BLOUNT, 1982. J. Org. Chem., 47, 3991-3993.

Monoclinic,  $P2_1$ ,  $a = 9.920$ ,  $b = 8.743$ ,  $c = 9.256$  Å,  $\beta = 117.02^\circ$ ,  $Z = 2$ . Cu

radiation,  $R = 0.033$  for 1008 reflexions.

The lactone ring and the epoxide rings of ivaxillin (Fig. 1) are fused *trans* to the ten-membered ring, and the C-11 methyl group is pseudoaxial and  $\beta$ . The  $\gamma$ -lactone ring is an envelope with C-7 as the flap. The C-4 and the C-10 methyl group lie  $\beta$  to the plane of the molecule.



Fig. 1. The ivaxillin molecule.

#### NEOPULCHELLIN

$C_{15}H_{22}O_4$

S. INAYAMA, K. HARIMAYA, H. HORI, T. KAWAMATA, T. OHKURA, H. NAKAMURA and Y. IITAKA, 1982. *Heterocycles*, **19**, 1801-1806.

Monoclinic,  $P2_1$ ,  $a = 8.308$ ,  $b = 10.494$ ,  $c = 7.921$  Å,  $\beta = 103.57^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.053$  for 921 reflexions.

The cycloheptane ring in the title compound (Fig. 1) assumes a boat conformation, the central plane consisting of atoms C(5), C(6), C(9) and C(10), and the "wing" planes consisting of C(6), C(7), C(8), C(9) and C(5), C(1) and C(10). The cyclopentane ring has a C(5)-envelope conformation while the  $\gamma$ -butyrolactone ring is virtually planar.

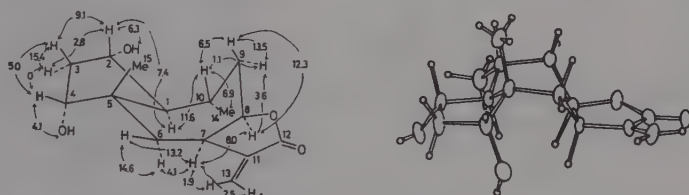


Fig. 1.  $^1H$ -NMR J-values for  $C_{15}H_{22}O_4$  (showing labelling) (left), and perspective view of the molecular structure (right).

#### 7,8-EPOXYHALOCHAMIGRENE

$C_{15}H_{23}Br_2C_{10}$

A. FURUSAKI, C. KATAYAMA, T. MATSUMOTO, M. SUZUKI, T. SUZUKI, H. KIKUCHI and E. KUROSAWA, 1982. *Bull. Chem. Soc. Jpn.*, **55**, 3398-3402.

Orthorhombic,  $P2_12_12_1$ ,  $a = 10.303$ ,  $b = 71.31$ ,  $c = 9.061$  Å,  $Z = 16$ . Cu radiation,  $R = 0.072$  for 4241 reflexions.

The four crystallographically independent molecules have almost the same geometries (Fig. 1). The six-membered ring with the epoxy group has a half-chair conformation, and the other ring has a chair conformation.

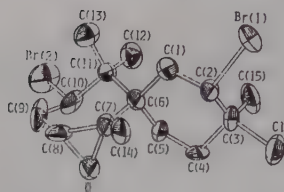


Fig. 1. A molecule of  $C_{15}H_{23}Br_2ClO$ .

#### MUROLENE DIHYDROCHLORIDE



A.-K. BORG-KARLSON, T. NORIN, A.-M. PILOTTI, T. STEFANIDES, A.-C. SÖDERHOLM and D. WIJEKON, 1982. *Acta Chem. Scand.*, **B36**, 137-139.

Monoclinic,  $P2_1$ ,  $a = 13.699$ ,  $b = 11.633$ ,  $c = 9.855$  Å,  $\beta = 95.08^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.135$  for 2107 reflexions.

The two molecules in the asymmetric unit are similar (Fig. 1) within the bounds of experimental error, and the bond lengths and angles of the two molecules appear to be normal in spite of the low accuracy. No discussion of the molecular geometry is attempted because of this low accuracy.

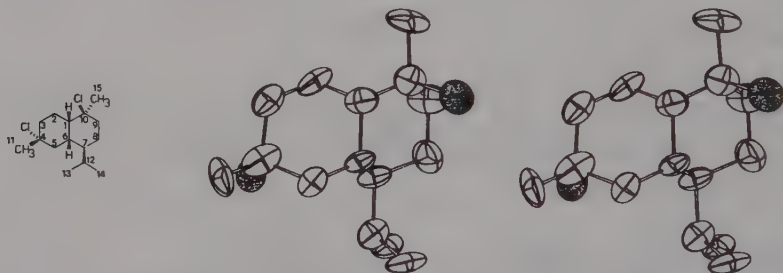
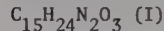


Fig. 1. Stereoscopic drawing of one of the independent molecules of  $C_{15}H_{24}Cl_2$ . The Cl atoms are shaded.

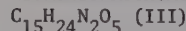
#### HUMULENE NITROSITE



#### DINITROHUMULENE



#### NITRONITRATOHUMULENE



D.K. MacALPINE, A.L. PORTE and G.A. SIM, 1982. *J. Chem. Soc. Perkin I*, 1385-1388.

I. Monoclinic,  $P2_1/n$ ,  $a = 6.484$ ,  $b = 18.139$ ,  $c = 14.294$  Å,  $\beta = 101.33^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.071$  for 1221 reflexions.

II. Monoclinic,  $P2_1/n$ ,  $a = 6.422$ ,  $b = 18.177$ ,  $c = 14.218$  Å,  $\beta = 100.69^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.042$  for 1617 reflexions.

III. Orthorhombic,  $Pc2_1n$ ,  $a = 7.665$ ,  $b = 11.908$ ,  $c = 18.170$  Å,  $Z = 4$ . Cu radiation,  $R = 0.052$  for 1610 reflexions.

Compounds I-III (Fig. 1) have a common ring conformation, which differs from that of humulene in the region of the NO, NO<sub>2</sub> and ONO<sub>2</sub> substituents. The C(3)-C(4) bond has a torsion angle of ca. -80° in I-III compared with 162° for the corresponding trans double bond in humulene. The bond angles in the eleven-membered ring indicate some degree of steric strain, C=C angles range from 121.5-129.3, and the C-C-C angles range from 105.8-116.5°.

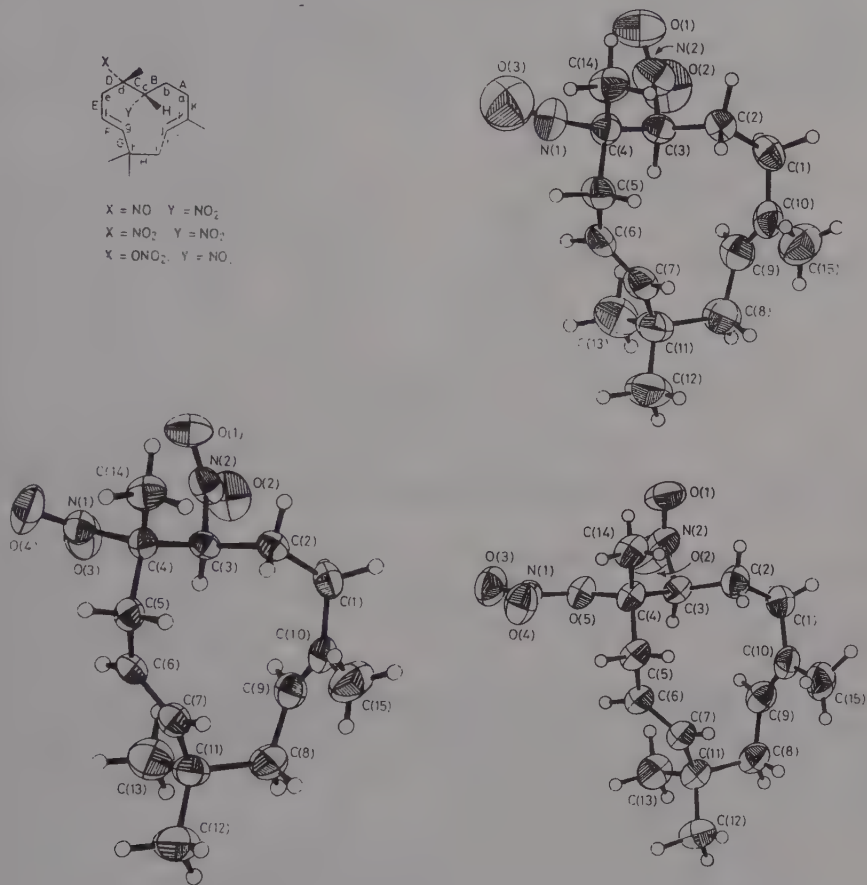
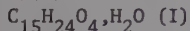
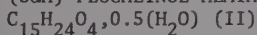


Fig. 1. Molecular skeletons of I-III (top left). Structure of C<sub>15</sub>H<sub>24</sub>N<sub>2</sub>O<sub>3</sub> (I) (top right), C<sub>15</sub>H<sub>24</sub>N<sub>2</sub>O<sub>4</sub> (II) (bottom left) and C<sub>15</sub>H<sub>24</sub>N<sub>2</sub>O<sub>5</sub> (III) (bottom right).

PLUCHEINOL HYDRATE



(3αH)-PLUCHEINOL HEMIHYDRATE



V. ZABEL, W.H. WATSON and M. SILVA, 1982. Acta Cryst., B38, 584-588.



I. Orthorhombic,  $P2_12_12_1$ ,  $a = 9.902$ ,  $b = 24.179$ ,  $c = 6.259$  Å,  $Z = 4$ . Cu radiation,  $R = 0.058$  for 1617 reflexions.

II. Monoclinic,  $P2_1$ ,  $a = 11.034$ ,  $b = 12.143$ ,  $c = 11.509$  Å,  $\beta = 95.83^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.050$  for 2712 reflexions.

The cyclohexane rings in I and II have chair conformations with the C(4) hydroxyl groups equatorial and are trans fused to cyclohexenone rings with 1,2-diplanar conformations. The C(3) hydroxyl group is equatorial in I and axial in II (Fig. 1). The two independent (3aH) molecules have slightly different conformations because of the extensive system of hydrogen-bond formation through the water molecules which also occurs in I.

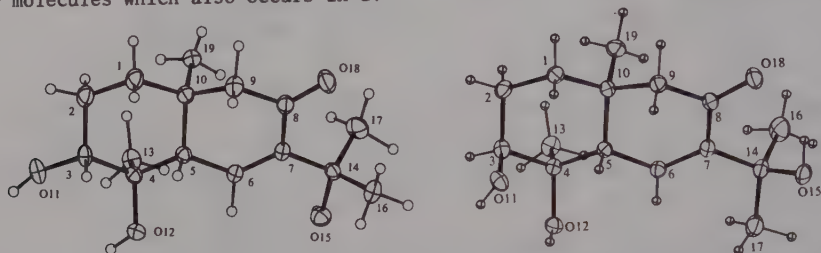


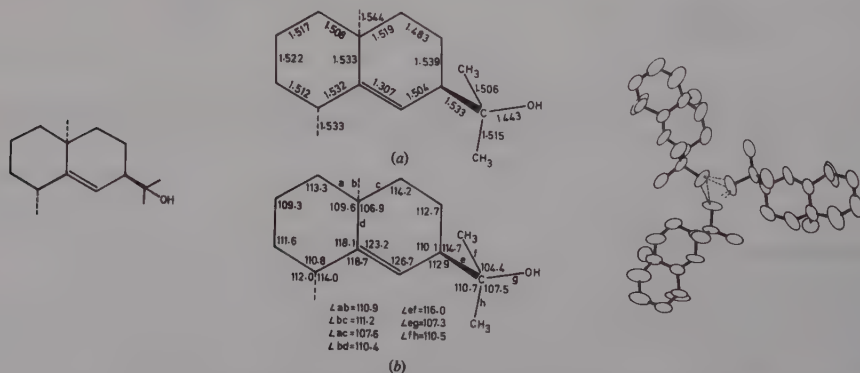
Fig. 1. Views of plucheinol (left) and (3aH)-plucheinol (right).

(+)-2-{(3R,6S,10R)-6,10-DIMETHYLBICYCLO[4.4.0]DEC-1-EN-3-YL}-2-PROPANOL (ROSIFOLIOL)  
 $C_{15}H_{26}O$

B. BEAGLEY, R.G. PRITCHARD, R. RAMAGE and I.A. SOUTHWELL, 1982. Acta Cryst., B38, 1391-1393.

Rhombohedral,  $R3$ ,  $a = 14.004$  Å,  $\alpha = 117.7^\circ$ ,  $Z = 3$ . Mo radiation,  $R = 0.053$  for 681 reflexions.

In the molecule (Fig. 1) the chair-half-chair ring system is strained due to the 1,3-diaxial methyl substituents (C...C 3.39 Å). The  $-C(CH_3)_2OH$  side chain adopts a staggered conformation with respect to ring bonds. The molecules form a hydrogen-bonding system in the crystal extending parallel to  $z$ . Adjacent molecules of the infinite chains are related by threefold screw axes and linked through their hydroxyl groups by hydrogen bonds of length 2.74 Å.



(±)-HIMACHALOL

 $C_{15}H_{26}O$ 

P. BHAN, S. DEV, L.S. BASS, B. TAGLE and J. CLARDY, 1982. *J. Chem. Research*, S, 344-345; M, 3473-3491.

Monoclinic,  $C2$ ,  $a = 21.473$ ,  $b = 8.549$ ,  $c = 16.288$  Å,  $\beta = 72.14^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.069$  for 1966 reflexions.

There are two molecules in the asymmetric unit. The configuration at C(7) (Fig. 1) was established by the X-ray analysis. Bond lengths ( $\sigma$  0.013-35 Å) and angles ( $\sigma$  0.7-2.2°) have acceptable values.

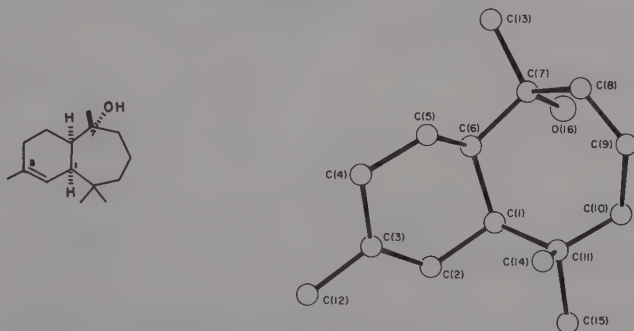


Fig. 1.  $C_{15}H_{26}O$ : the molecular skeleton and structure.

VERBESINDIOL HYDRATE

 $C_{15}H_{28}O_2 \cdot 0.25(H_2O)$ 

W. HERZ, N. KUMAR and J.F. BLOUNT, 1982. *J. Org. Chem.*, 47, 1785-1786.

Orthorhombic,  $P2_12_12_1$ ,  $a = 9.917$ ,  $b = 23.081$ ,  $c = 26.535$  Å,  $Z = 16$ . Cu radiation,  $R = 0.045$  for 3716 reflexions.

The four independent molecules in the asymmetric unit have nearly the same conformations. The stereochemistry is shown in Fig. 1.

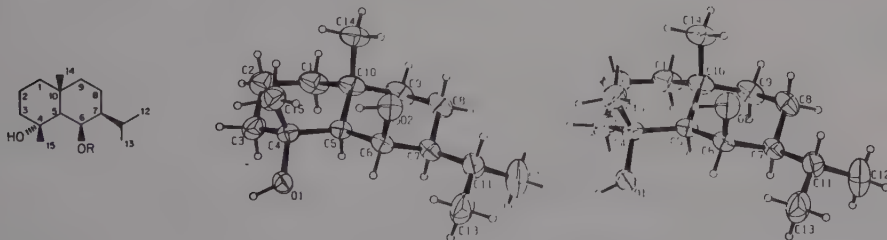


Fig. 1. A stereoview of one of the verbesindiol molecules.

IRAZUNOLIDE

 $C_{17}H_{18}O_5$ 

C. HASBUN, M.A. CALVO, L.J. POVEDA, A. MALCOLM, T.J. DELORD, S.F. WATKINS, F.R. FRONCZEK and N.H. FISCHER, 1982. *J. Nat. Prod.*, 45, 749-753.

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.793$ ,  $b = 9.394$ ,  $c = 21.439$  Å,  $Z = 4$ . Mo radiation,  $R = 0.0451$  for 712 reflexions.

The structure of this sesquiterpene lactone was determined to be as shown in Fig. 1.

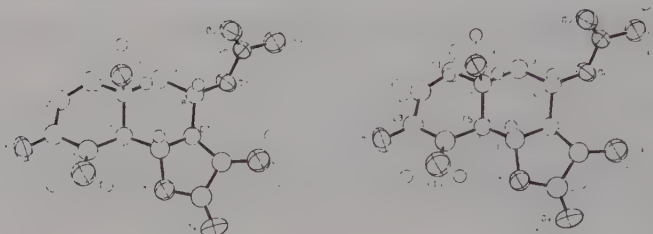
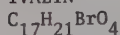


Fig. 1. A stereoview of irazumolide.

# IVALIN



J. COETZER, G.J. KRUGER and D.C. LEVENDIS, 1982. S. Afr. J. Chem., 35, 103-104.

Monoclinic,  $P2_1$ ,  $a = 11.65$ ,  $b = 6.96$ ,  $c = 11.00$  Å,  $\beta = 115.95^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.052$  for 1074 reflexions.

Rings A and B are both in the chair conformations,  $^1C_4$ , typical for an unstrained trans-fused ring junction. The cis-fused  $\alpha$ -methylene- $\gamma$ -lactone ring has the twist-chair conformation,<sup>5</sup>T. It exerts little influence on the shape of the rest of the molecule, which is essentially that of trans-8,9-dimethyl-3-decalone.

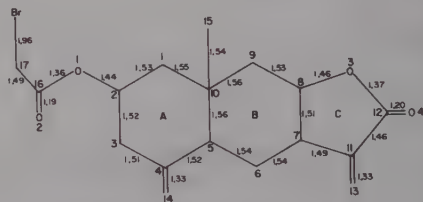
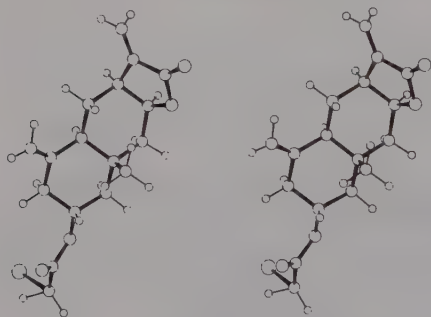


Fig. 1. A stereoview of ivalin and bond lengths.

# AMBLIODIOL



W. HERZ and J.F. BLOUNT, 1982. J. Org. Chem., 47, 1594-1595.

Trigonal,  $P3_1$ ,  $a = 9.682$ ,  $c = 15.549$  Å,  $Z = 3$ . Cu radiation,  $R = 0.054$  for 1397 reflexions.

The analysis shows that the C11 hydroxyl group is  $\beta$  (Fig. 1). The cycloheptane ring is a twist chair whose twofold axis of symmetry passes through C-6 and the midpoint of the C-9,C-10 bond.  $E_2$ , the deviation of the ring from  $C_2$  symmetry, is only  $8^\circ$ . The two five-membered rings are attached to the cycloheptane ring in the C-5(e), C-1(e) and the C-7(e), C-8(e) positions, respectively. The cyclopentenone is an envelope with C-5 as the flap, and the  $\gamma$ -lactone ring is also an envelope with C-7 as the flap.

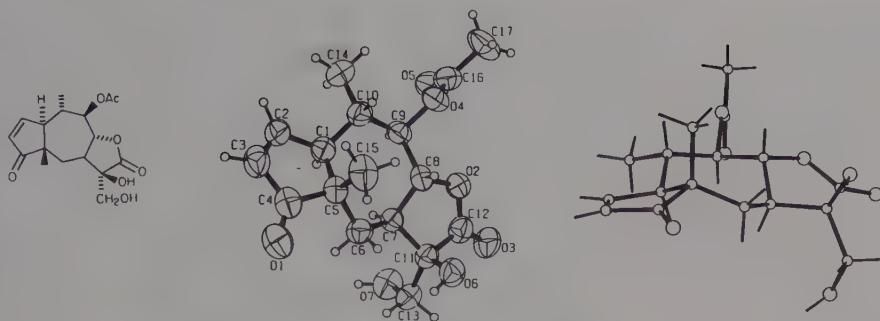


Fig. 1. Amblyodiol.

# PHOTOSANTONIN



R.I. SHELDON, C.G. SHAEFER, J. GULBIS, J.R. RUBLE, A.W. BURGSTAHLER and B. LEE, 1982. *Acta Cryst.*, **B38**, 649-651.

Orthorhombic,  $P2_12_12_1$ ,  $a = 8.767$ ,  $b = 8.773$ ,  $c = 21.101$  Å,  $Z = 4$ . Mo radiation,  $R = 0.056$  for 1986 reflexions (at 242 K).

The dihedral angle  $C(7)-C(1)-C(2)-C(10)$  of the diene (Fig. 1) is  $64.3(4)^\circ$  and the  $C(1)-C(2)$  bond length is  $1.505(2)$  Å. The average endocyclic angle in the six-membered ring is  $56.7^\circ$ .

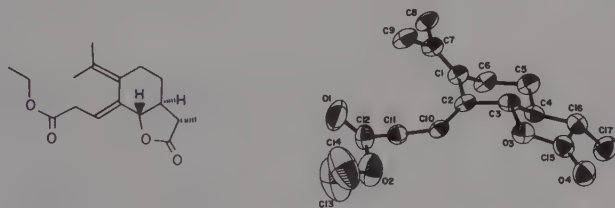
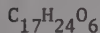


Fig. 1. The photosantonin molecule.

# CHAMISSONOLIDE



M. WIEBCKE, J. KRESKEN, D. MOOTZ and G. WILLUHN, 1982. *Tetrahedron*, **38**, 2709-2714.

Orthorhombic,  $P2_12_12_1$ ,  $a = 8.996$ ,  $b = 9.807$ ,  $c = 18.890$  Å,  $Z = 4$ . Mo radiation,  $R = 0.051$  for 1457 reflexions.

The configuration and conformation of chamissonolide, a new pseudoguaianolide, were established as shown in Fig. 1. Molecules are linked by O-H...O hydrogen bonds.



The analysis confirms the basic skeletal arrangement and relative stereochemistry of all chiral centres and establishes the epoxide at C11-C13 to be  $\alpha$ -oriented (Fig. 1). The dihedral angle formed by the furenone ring and the plane of the double bond (C3/C4/C5/C6/C15) is  $86.1^\circ$ . The angle between the planes of the 2,3-double bond and 4,5-double bond of nearly  $90^\circ$  does not allow extended conjugation of the C-1 carbonyl chromophore and is the reason for the low wavelength uv absorption near 260 nm.

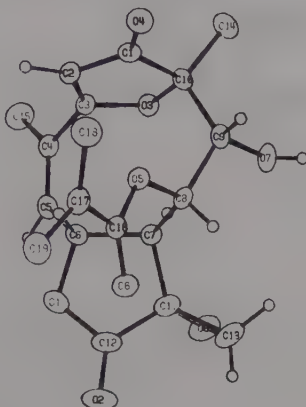
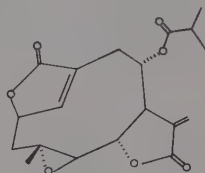


Fig. 1. A view of C<sub>19</sub>H<sub>20</sub>O<sub>8</sub>.

#### DIHYDROELEPHANTOPIN

C<sub>19</sub>H<sub>22</sub>O<sub>7</sub>



W.H. WATSON, V. ZABEL, T.J. MABRY and G. YABUTA, 1982. *Acta Cryst.*, B38, 511-514.

Orthorhombic, P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>,  $a = 10.952$ ,  $b = 25.642$ ,  $c = 6.386$  Å,  $Z = 4$ . Cu radiation,  $R = 0.061$  for 1568 reflexions.

The structure of the molecule is as shown. The ten-membered ring has a chair-chair conformation and the two five-membered rings are flattened envelopes.

#### ACROPTILIN

C<sub>19</sub>H<sub>23</sub>ClO<sub>7</sub>

K.L. STEVENS and R.Y. WONG, 1982. *Cryst. Struct. Comm.*, 11, 949-954.

Orthorhombic, P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>,  $a = 11.450$ ,  $b = 19.676$ ,  $c = 8.269$  Å,  $Z = 4$ . Cu radiation,  $R = 0.0574$  for 2708 reflexions.

The absolute configuration is shown in Fig. 1, and the 3-OH and epoxide functions are trans. The cyclopentane ring is puckered such that C1 is below the plane of C2, C3, C4 and C5. The 7-membered ring assumes a conformation in which C9 is folded up bringing H2C9 and HC6 within 2.433 Å of each other.



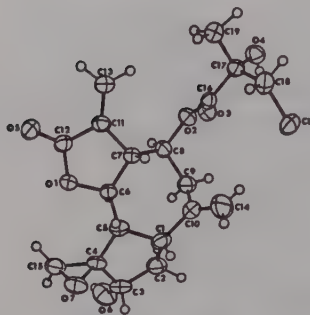


Fig. 1. A view of acroptitin.

## LYCHNOPHOROLIDE A

 $C_{20}H_{22}O_7$ 

P.W. LE QUESNE, M.D. MENACHERY, M.P. PASTORE, C.J. KELLEY, T.F. BRENNAN, K.D. ONAN, R.F. RAFFAUF and C.M. WEEKS, 1982. *J. Org. Chem.*, **47**, 1519-1521.

Monoclinic,  $P2_1$ ,  $a = 9.343$ ,  $b = 11.808$ ,  $c = 8.500$  Å,  $\beta = 95.57^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.055$  for 948 reflexions.

In the molecule (Fig. 1), the C(6),C(7) trans-fused  $\alpha$ -methylene  $\gamma$ -lactone is quite planar, with the largest deviation from the best mean plane through atoms C(6), C(7), C(11), C(12), and O(2) being 0.02 Å. The conformation of the cyclo-decadienone ring is essentially dictated by the  $\beta$ -side ether linkage between C(3) and C(10). Not only does this bridge require the near-planarity of atoms C(1), C(2), C(3), and C(10) but it also causes a severe transannular interaction between O(4) and H(7).

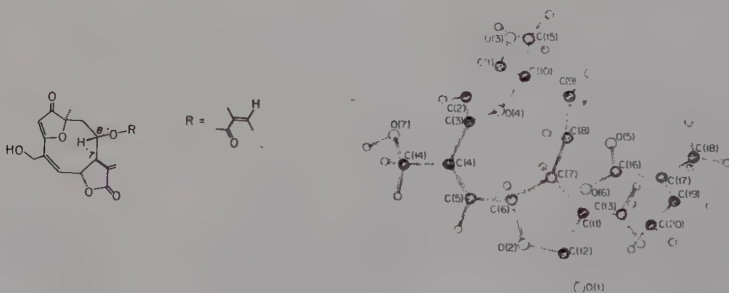


Fig. 1. Lychnophorolide A.

## 88-ANGELOYLOXYMAXIMILIANIN

 $C_{20}H_{26}O_5$ 

W.H. WATSON and V. ZABEL, 1982. *Acta Cryst.*, **B38**, 1608-1610.

Monoclinic,  $P2_1$ ,  $a = 10.116$ ,  $b = 11.979$ ,  $c = 7.787$  Å,  $\beta = 92.30^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.056$  for 1716 reflexions.

The five- and seven-membered carbocyclic rings are cis fused while the lactone ring has a trans ring juncture (Fig. 1). The two five-membered rings have flattened envelope conformations while that of the seven-membered ring is a twist chair. Molecules are linked by O-H...O (2.889(3) Å) hydrogen bonds.

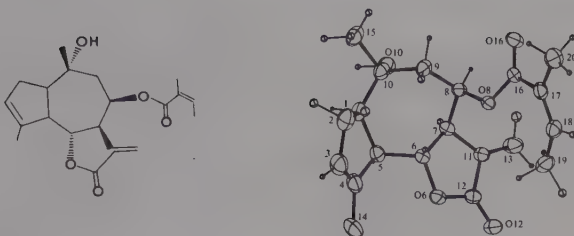


Fig. 1. The 8 $\beta$ -angeloyloxymaximilianin molecule.

# ARGOPHYLLIN-A

$C_{20}H_{28}O_7$  (I)

(8S)-METHACRYLOXY-1,10;4,5-DIEPOXYCOSTUNOLIDE

$C_{19}H_{24}O_6$  (II)

W.H. WATSON and V. ZABEL, 1982. *Acta Cryst.*, B38, 834-838.

I. Orthorhombic,  $P2_12_12_1$ ,  $a = 11.506$ ,  $b = 17.273$ ,  $c = 9.999$  Å,  $Z = 4$ . Cu radiation,  $R = 0.060$  for 1760 reflexions.

II. Orthorhombic,  $P2_12_12_1$ ,  $a = 12.296$ ,  $b = 18.595$ ,  $c = 7.735$  Å,  $Z = 4$ . Cu radiation,  $R = 0.068$  for 1779 reflexions.

In both I and II (Fig. 1) the ten-membered rings adopt approximate chair-chair conformations, but the torsion angles differ by an average of  $20^\circ$ . The germacradiene skeleton normally contains C(1)-C(10) and C(4)-C(5) trans double bonds. In II both double bonds are epoxidized, while in I C(1)-C(10) is epoxidized and C(4)-C(5) is saturated, thus transmitting conformational changes around the ten-membered ring. The C(8) side chain in I is  $\beta$  while that in II is  $\alpha$ .

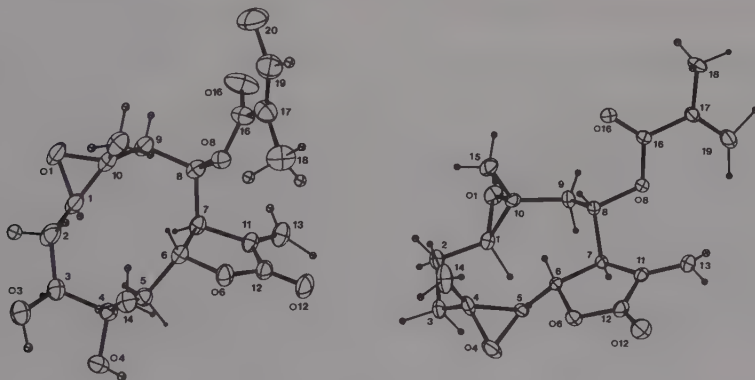


Fig. 1. Perspective views of molecules of  $C_{20}H_{28}O_7$  (I) (left) and  $C_{19}H_{24}O_6$  (II) (right).

# EPOXYINEUPATOROLIDE B1 BROMOHYDRIN

$C_{20}H_{29}BrO_7$

N.C. BARUAH, R.N. BARUAH, R.P. SHARMA, J.N. BARUAH, W. HERZ, K. WATANABE and J.F. BLOUNT, 1982. *J. Org. Chem.*, 47, 137-140.

The absolute configuration of the germacranolides ineupatorolides A and B has been established from the analysis by the anomalous dispersion method of a bromohydrin derivative (Fig. 1).

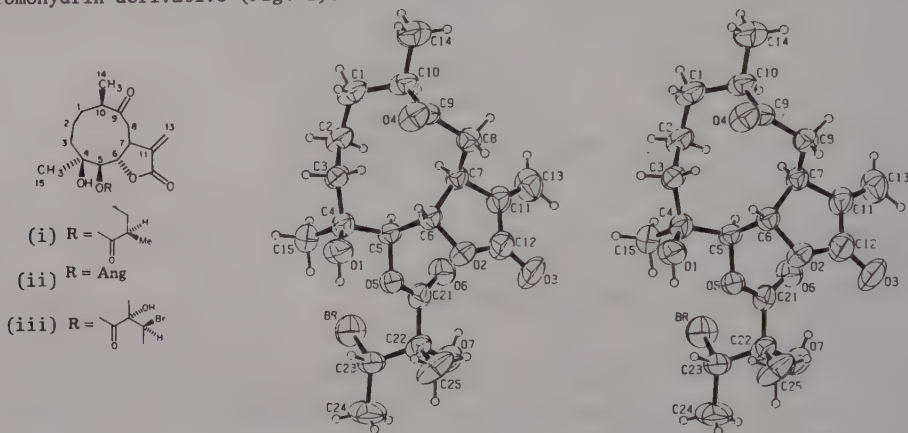


Fig. 1. Formulae of (i) ineupatorolide A, (ii) ineupatorolide B and (iii) the bromohydrin derivative  $C_{20}H_{29}BrO_7$ , and a stereoview of the last, showing the correct absolute stereochemistry.

$$\text{C}_{20}\text{H}_{30}\text{O}_7$$

Monoclinic,  $P2_1$ ,  $a = 8.528$ ,  $b = 10.421$ ,  $c = 11.530$  Å,  $\beta = 92.76^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.036$  for 1344 reflexions.

The C11 hydroxyl group is  $\beta$  (Fig. 1) and the cycloheptane ring closely approximates a twist boat, whose  $C_2$  axis passes through C-10 and the midpoint of the C-6,C-7 bond.  $\Sigma_2$ , the deviation of the ring from  $C_2$  symmetry, is only  $11^\circ$ . The two five-membered rings are attached to the cycloheptane ring in the C-5(e), C-1(e), and the C-7(e), C-8(e) positions, respectively. The cyclopentanone and the lactone rings are very slightly distorted envelopes with C-5 and C-7 as the respective flaps.

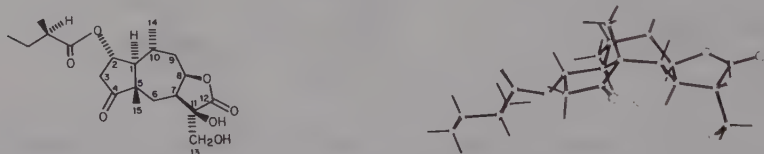


Fig. 1. Florigrandin.

$$\text{C}_{21}\text{H}_{26}\text{N}_4\text{O}_4$$

M.A. RUSSELL, G.A. SIM and D.N.J. WHITE, 1982. J. Chem. Soc. Perkin II, 245-247.

Monoclinic,  $P2_1/c$ ,  $a = 12.619$ ,  $b = 7.290$ ,  $c = 23.422$  Å,  $\beta = 98.06^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.046$  for 3222 reflexions.

All three double bonds of this sesquiterpenoid ketone from "Zingiber zerumbet" are shown to have a trans configuration (Fig. 1). The cycloundecatriene has a conformation with approximate  $C_2$  symmetry, the significant departures from this being the torsion angles around C(1)-C(2) and C(1)-C(11) of  $-26$  and  $-52^\circ$  respectively. The large value for the latter arises from interaction between 11-H and the hydrazone NH (H...H 2.31 Å). The four ring C-C(sp<sup>3</sup>)-C angles range from  $106.3$  to  $113.3$  and the six C-C=C angles range from  $118.3$  to  $129.4^\circ$  indicating some degree of steric strain.

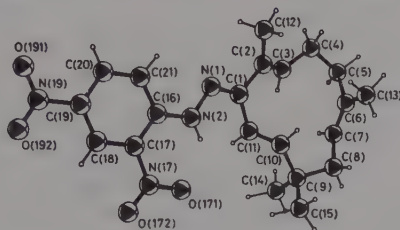
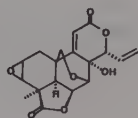
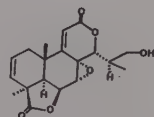


Fig. 1. Molecular structure of  $C_{21}H_{26}N_4O_4$ .

SALIGNONE-A  
 $C_{18}H_{18}O_7$  (I)



SALIGNONE-H  
 $C_{19}H_{22}O_6$  (II)



W.H. WATSON, V. ZABEL, M. SILVA and M. BITTNER, 1982. *Acta Cryst.*, **B38**, 588-592.

I. Monoclinic,  $P2_1$ ,  $a = 10.469$ ,  $b = 11.337$ ,  $c = 6.764$  Å,  $\beta = 104.70^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.034$  for 1080 reflexions.

II. Monoclinic,  $P2_1$ ,  $a = 11.921$ ,  $b = 7.686$ ,  $c = 9.848$  Å,  $\beta = 112.02^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.056$  for 1514 reflexions.

In both compounds the A and B rings are trans-fused with A having the 1,2-diplanar conformation. The epoxide group in II gives the B ring a distorted 1,2-diplanar conformation while in I, with fused axial substituents, this ring has a twisted boat conformation. The  $\alpha,\beta$  unsaturated  $\delta$ -lactones are in distorted 1,3-diplanar conformations while the  $\gamma$ -lactones have flattened envelope conformations.

PINNATAL  
 $C_{20}H_{18}O_5$

K.C. JOSHI, P. SINGH, S. TANEJA, P.J. COX and R.A. HOWIE, 1982. *Tetrahedron*, **38**, 2703-2708.

Trigonal,  $P3_1$ ,  $a = 7.836$ ,  $c = 23.45$  Å,  $Z = 3$ . Mo radiation,  $R = 0.035$  for 1471 reflexions.

The structure and relative stereochemistry are shown in Fig. 1. The rings have the following conformations: quinone, boat; cyclohexene, twist-boat; cyclopentane, distorted C7 envelope; dihydropyran, boat; oxepane, chair.

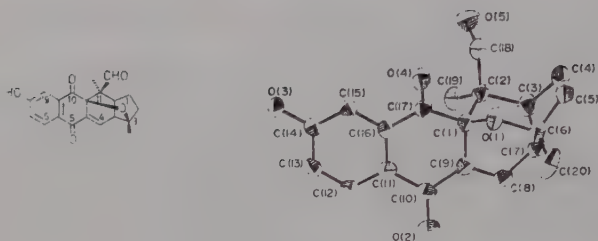
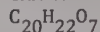


Fig. 1. The pinnatal molecule.

#### CHAMAEDROXIDE



L. EGUREN, A. PERALES, J. FAYOS, B. RODRIGUEZ, G. SAVONA and P. PIOZZI, 1982. J. Org. Chem., 47, 4157-4160.

Orthorhombic,  $P2_12_12_1$ ,  $a = 30.139$ ,  $b = 11.3698$ ,  $c = 10.3159$  Å,  $Z = 8$ . Cu radiation,  $R = 0.061$  for 2038 reflexions.

The analysis establishes the absolute configuration shown in Fig. 1. There are two independent molecules linked by O-H...O hydrogen bonds (2.746 and 2.964(5) Å). Ring C is in an envelope conformation with the flap atom C11 at the C10 side. Rings A and B are deformed boats, with flaps at C3, C10 and C7.

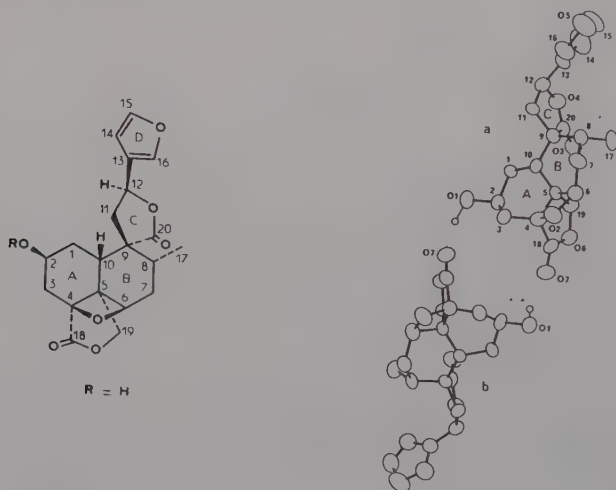


Fig. 1. The chamaedroxide structure.

#### PUMILIN



J.D. KORP, I. BERNAL, N.H. FISCHER, C. LEONARD, I.-Y. LEE and N. LeVAN, 1982. J. Heterocyclic Chem., 19, 181-187.

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.065$ ,  $b = 13.652$ ,  $c = 19.586$  Å,  $Z = 4$ . Mo radiation,  $R = 0.039$  for three-dimensional data.

The analysis establishes the structure of pumilin (Fig. 1). The cycloheptene ring is in chair conformation with the lactone ring a half-chair and the cyclopentenone ring essentially planar. Molecules are linked into chains by O-H...O hydrogen bonds.

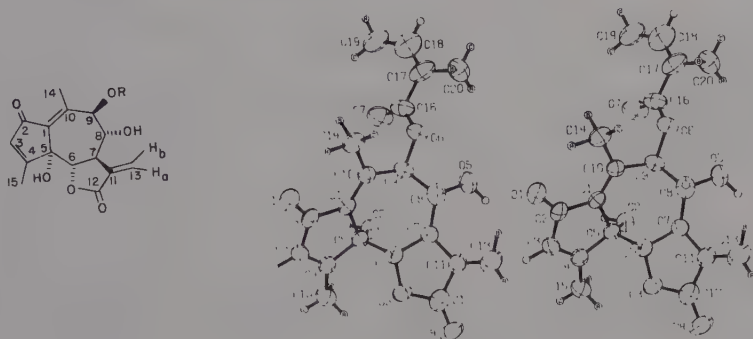


Fig. 1. The pumilin molecule ( $R = \text{CH}_3\text{-CH=CCH}_3\text{-CO}$ ) and a stereoview.

#### LOPHODIONE

$\text{C}_{20}\text{H}_{24}\text{O}_4$

M.M. BANDURRAGA, B. MCKITTRICK, W. FENICAL, E. ARNOLD and J. CLARDY, 1982. Tetrahedron, 38, 305-310.

Monoclinic,  $P2_1$ ,  $a = 6.329$ ,  $b = 13.925$ ,  $c = 10.438 \text{ \AA}$ ,  $\beta = 105.20^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.053$  for 1581 reflexions.

The analysis defined the stereochemistry as shown in Fig. 1. The C(8)-C(9) double bond has E-configuration and the C(11)-C(12) configuration is Z.

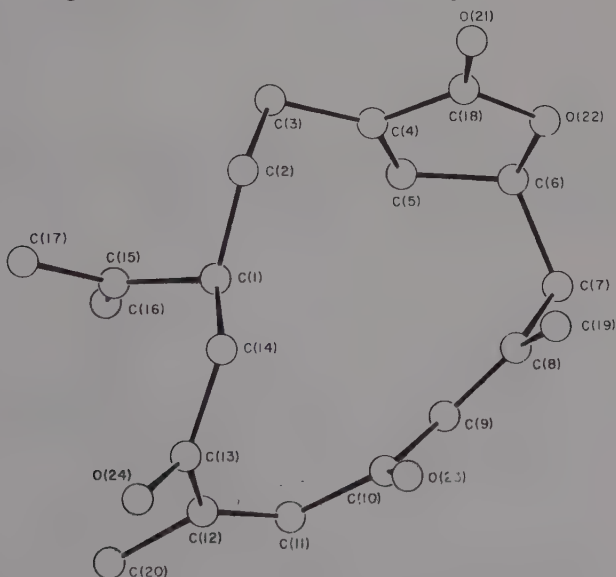


Fig. 1. The lophodione molecule.



## 4-KETO-all-trans-RETINAL



A. GIEREN and V. LAMM, 1982. *Z. Naturforsch.*, **37b**, 1612-1622.

Monoclinic,  $P2_1/n$ ,  $a = 14.761$ ,  $b = 8.292$ ,  $c = 15.210$  Å,  $\beta = 102.40^\circ$ ,  $D_m = 1.09$ ,  $Z = 4$ . Cu radiation,  $R = 0.038$  for 1211 reflexions.

The cyclohexanone ring (Fig. 1) adopts a half-boat conformation and the connection of this ring and the conjugated polyene chain is S-cis (the C(5)C(6)-C(7)C(8) torsion angle is  $56^\circ$ ).

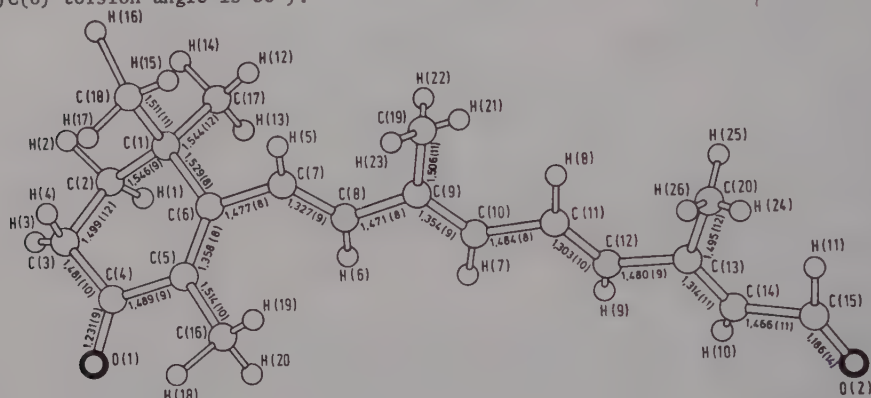


Fig. 1. Bond lengths (Å) and e.s.d.'s in  $\text{C}_{20}\text{H}_{26}\text{O}_2$ .

## RIOLOZATRIONE



W.H. WATSON, V. ZABEL and X.A. DOMINGUEZ, 1982. *Acta Cryst.*, **B38**, 689-691.

Orthorhombic,  $P2_12_12_1$ ,  $a = 11.404$ ,  $b = 21.090$ ,  $c = 7.506$  Å,  $Z = 4$ . Cu radiation,  $R = 0.053$  for 1586 reflexions.

In the riolozatrione molecule (Fig. 1) the two five-membered rings have flattened envelope conformations with the keto-substituted ring approaching planarity. The angle between the C(1)C(3)C(4)C(8) and C(4)C(5)C(7)C(8) planes is  $173.5^\circ$ . The six-membered ring has a 1,2 diplanar conformation with C(9) deviating from the ring plane. The C(2)-C(19) distance is  $1.609(5)$  Å although C(19) exhibits large thermal motion.

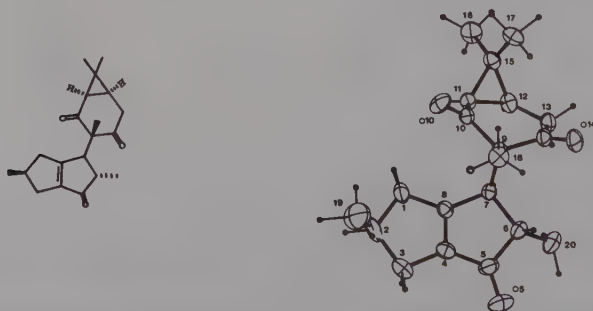
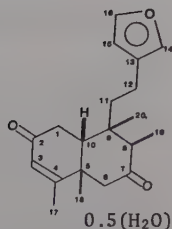
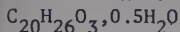


Fig. 1. The riolozatrione molecule.

## EREMONE HEMIHYDRATE

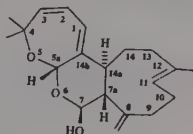


S.J. JOLAD, J.J. HOFFMANN, K.H. SCHRAM, J.R. COLE, M.S. TEMPESTA and R.B. BATES, 1982. *J. Org. Chem.*, **47**, 1356-1358.

Monoclinic,  $P2_1$ ,  $a = 6.890$ ,  $b = 12.954$ ,  $c = 9.984$  Å,  $\beta = 102.44^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.070$  for 969 reflexions.

Ring A has a 1,2-diplanar cyclohexene conformation and ring B is in chair form. In the side chain, C(12) is within  $1^\circ$  of being anti to C(20), and C(13) is  $3^\circ$  from being anti to C(9). The C(11)-C(12)-C(13)-C(14) torsion angle is  $42^\circ$ .

(5aR\*, 7R\*, 7aS\*, 11E, 14aR\*)-4,4,12-TRIMETHYL-8-METHYLENE-4,5,7,7a,8,9,10,13,14,14a-DECAHYDROCYCLONONA(1',2':4,5)PYRANO[2,3-b]OXEPIN-7-OL

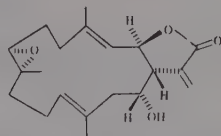


B.F. BOWDEN, J.C. COLL, E. DITZEL, S.J. MITCHELL and W.T. ROBINSON, 1982. *Aust. J. Chem.*, **35**, 997-1002.

Monoclinic,  $P2_1$ ,  $a = 12.812$ ,  $b = 8.265$ ,  $c = 8.264$  Å,  $\beta = 100.35^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.060$  for 1268 reflexions.

The structure of the diterpene was established as that shown above.

(1R,2R,3E,7R,8R,11E,14R)-7,8-EPOXY-14-HYDROXYCEMBRA-3,11,15-TRIEN-17,2-OLIDE

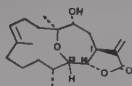


K.P. BURNS, R. KAZLAUSKAS, P.T. MURPHY, R.J. WELLS and P. SCHONHOLZER, 1982. *Aust. J. Chem.*, **35**, 85-94.

Monoclinic,  $P2_1$ ,  $a = 17.727$ ,  $b = 11.104$ ,  $c = 9.783$  Å,  $\beta = 92.89^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.0586$  for 2419 reflexions.

The structure analysis confirmed the structure shown above.

(13αH,14βH)-JEUNICIN



A.J. WEINHEIMER, J.A. MATSON, M. POLING and D. VAN DER HELM, 1982. *Acta Cryst.*, **B38**, 580-583.

Orthorhombic,  $P2_12_12_1$ ,  $a = 8.921$ ,  $b = 18.076$ ,  $c = 22.647$  Å, ( $a = 8.962$ ,  $b = 18.30$ ,  $c = 22.96$  Å at 295 K),  $D_m = 1.184$ ,  $Z = 8$ . Cu radiation,  $R = 0.063$  for 4191 reflexions (at 113 K).

The molecule, an epimer of jeunicin, has the half-chair,  $\gamma$ -lactone ring trans-fused to the 14-membered cembrane ring. The overall conformation of the two independent molecules is similar although the angles between the least-squares planes for the cembrane and  $\gamma$ -lactone rings are  $18.6$  and  $9.1^\circ$  with the r.m.s. deviation of atoms from the  $\gamma$ -lactone ring plane  $0.151$  and  $0.145$  Å. The molecules are linked by weak O-H...O hydrogen bonds.

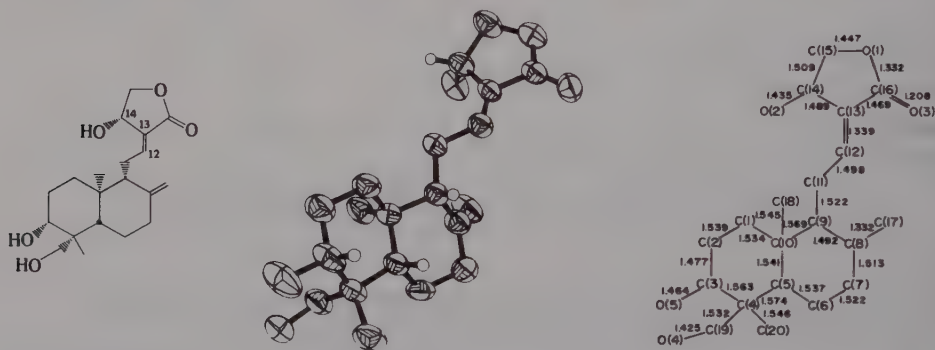
#### ANDROGRAPHOLIDE

$C_{20}H_{30}O_5$

A.B. SMITH, B.H. TODER, P.J. CARROLL and J. DONOHUE, 1982. *J. Cryst. Spectrosc. Res.*, **12**, 309-319.

Monoclinic,  $P2_1$ ,  $a = 6.550$ ,  $b = 8.005$ ,  $c = 17.991$  Å,  $\beta = 97.36^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.073$  for 1679 reflexions.

The analysis establishes the structure and stereochemistry of this natural product as shown in Fig. 1. All three OH groups are involved in hydrogen bonding.



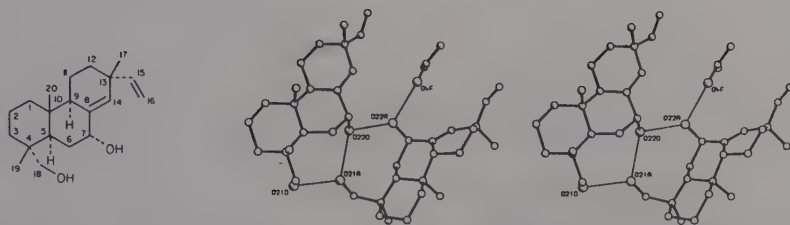
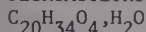


Fig. 1. The C<sub>20</sub>H<sub>32</sub>O<sub>2</sub> molecule and a stereopair showing the hydrogen bonding with acetone.

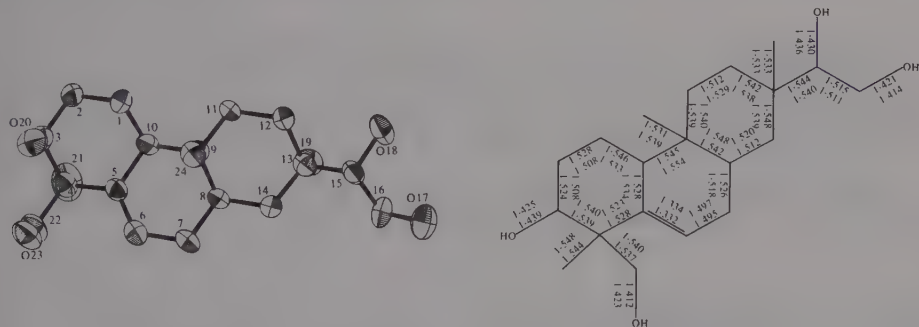
# JESROMOTETROL HYDRATE



V. ZABEL, W.H. WATSON, X.A. DOMÍNGUEZ, C. CISNEROS, E. GUAJARDO and R. VILLARREAL, 1982. *Acta Cryst.*, B38, 295-298.

Monoclinic, P2<sub>1</sub>,  $a = 26.48$ ,  $b = 6.622$ ,  $c = 11.550$  Å,  $\beta = 104.67^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.048$  for 3373 reflexions.

Bond lengths (Fig. 1) and angles are similar in the two crystallographically independent molecules. The three six-membered rings exist in flattened-chair, half-chair and chair conformations respectively, with a trans B/C ring juncture. The molecules are held together in the crystal by a network of hydrogen bonds involving water molecules.



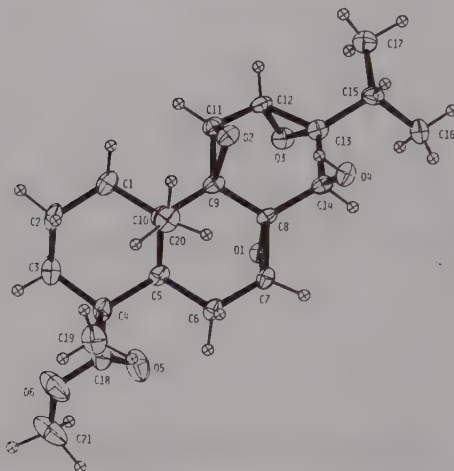
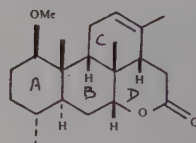


Fig. 1. A view of the trepoxide  $C_{21}H_{32}O_3$ .

(1 $\beta$ ,9 $\beta$ )-1-METHOXPICRAS-12-EN-16-ONE  
 $C_{21}H_{32}O_3$



R.C. HALTIWANGER and P.A. GRIECO, 1982. *Acta Cryst.*, B38, 694-696.

Monoclinic,  $P2_1/n$ ,  $a = 8.522$ ,  $b = 22.773$ ,  $c = 10.190$  Å,  $\beta = 111.32^\circ$ ,  $D_m = 1.22$ ,  $Z = 4$ . Mo radiation,  $R = 0.056$  for 1713 reflexions.

Rings A and B have chair configurations while C and D have envelope-like configurations. There are no unusual bond lengths or angles.

METHYL ent-15 $\beta$ -HYDROXYTRACHYLOBAN-19-OATE  
 $C_{21}H_{32}O_3$

G. FERGUSON, R. McCRINDLE, S.T. MURPHY and M. PARVEZ, 1982. *J. Chem. Research*, S, 200-201; M, 2009-2033.

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.634$ ,  $b = 10.190$ ,  $c = 23.613$  Å,  $Z = 4$ . Mo radiation,  $R = 0.041$  for 1445 reflexions.

Rings A and B of the molecule (Fig. 1) have slightly flattened chair conformations, rings C (C(8), C(9), C(11-14)) and D (C(8), C(9), C(11), C(12), C(16), C(15)) twisted boat forms and ring E (C(8), C(15), C(16), C(13), C(14)) a C(8) envelope conformation. The molecular dimensions have acceptable values: C(sp<sup>3</sup>)-C(sp<sup>3</sup>) 1.492(6)-1.502(5), C(sp<sup>3</sup>)-C(sp<sup>2</sup>) 1.532(5), C=O 1.204(5), C(sp<sup>2</sup>)-O 1.330(5), C(sp<sup>3</sup>)-O 1.426, 1.446(5), C-H 0.85-1.14(4) Å. The molecules are linked by OH...O hydrogen bonds to form spirals along  $b$  (O(1)...H(O3) 1.93 Å).

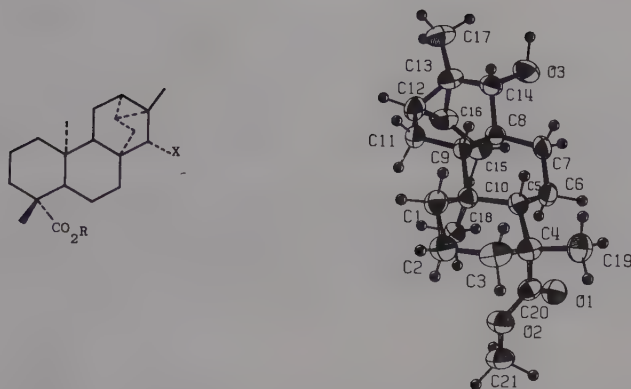


Fig. 1.  $C_{21}H_{32}O_3$ : the molecular skeleton ( $R = Me$ ,  $X = OH$ ) and structure.

#### TAFRICANIN A

$C_{22}H_{25}ClO_8$

J.R. HANSON, D.E.A. RIVETT, S.V. LEY and D.J. WILLIAMS, 1982. J. Chem. Soc. Perkin I, 1005-1008.

Monoclinic,  $P2_1$ ,  $a = 11.194$ ,  $b = 9.123$ ,  $c = 11.246$  Å,  $\beta = 111.66^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.032$  for 1465 reflexions. [The x coordinate for C(19) should be 0.2682.]

The absolute configuration of this chlorine containing diterpenoid (Fig. 1) is determined in addition to the molecular structure. Both rings A and B have the normal chair conformation. There is an intramolecular hydrogen bond (2.62 Å) between the 4-hydroxyl group and the 3-carbonyl group, and there is also a short non-bonded intramolecular contact (2.89 Å) between this 4-hydroxyl oxygen and the ether oxygen of the 19-acetoxy function.

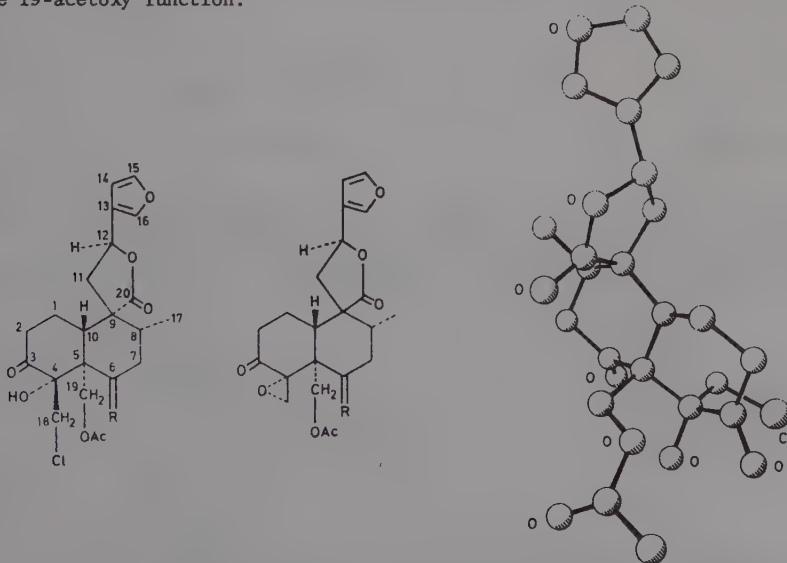


Fig. 1. Molecular skeleton ( $R = O$ ) and structure and absolute configuration of Tafricanin A.



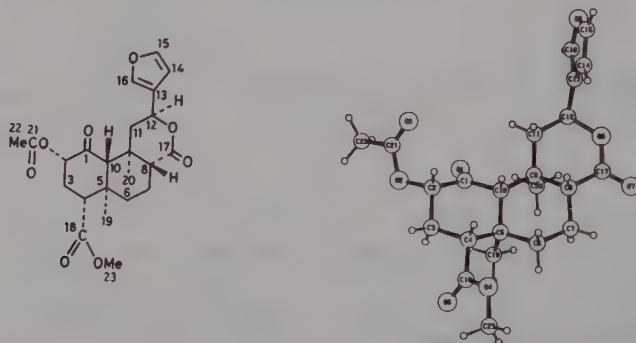
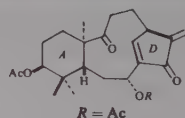
## SALVINORIN

 $C_{23}H_{28}O_8$ 

A. ORTEGA, J.F. BLOUNT and P.S. MANCHAND, 1982. J. Chem. Soc. Perkin I, 2505-2508.

Orthorhombic,  $P2_12_12_1$ ,  $a = 6.368$ ,  $b = 11.338$ ,  $c = 30.710$  Å,  $Z = 4$ . Cu radiation,  $R = 0.052$  for 1518 reflexions.

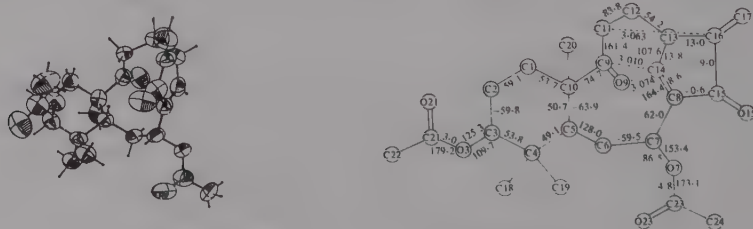
Salvinorin, from "Salvia divinorum", is shown to be a trans-neoclerodane diterpene. Bond lengths around the lactone ring are C(8)-C(9) 1.548, C(9)-C(11) 1.534, C(11)-C(12) 1.528, C(12)-O(6) 1.468, and C(17)-C(8) 1.500 and the carbonyl bond C(17)-O(7) is 1.199 Å. The bond angles around the lactone function are C(12)-O(6)-C(17) 123.9, C(8)-C(17)-O(6) 118.4, O(7)-C(17)-O(6) 117.0 and O(7)-C(17)-C(8) 124.4°.

Fig. 1. Molecular skeleton and structure of  $C_{23}H_{28}O_8$ .ent-9,15-DIOXO-8,9-SECO-14,16-KAURADIENE-3 $\alpha$ ,7 $\beta$ -DIOL DIACETATE (SHIKOCCIN MONOACETATE)  
 $C_{24}H_{32}O_6$ 

T. TAGA, K. OSAKI, N. ITO and E. FUJITA, 1982. Acta Cryst., B38, 2941-2944.

Orthorhombic,  $P2_12_12_1$ ,  $a = 13.218$ ,  $b = 19.053$ ,  $c = 9.205$  Å,  $Z = 4$ . Cu radiation,  $R = 0.082$  for 1679 reflexions.

In the molecule (Fig. 1) the A ring has a distorted chair form, and the ten-membered B/C ring has approximate  $C_2$  symmetry. Intramolecular non-bonding contacts of 3.010(12) and 3.074(12) Å are observed for C(9)...C(14) and O(9)...C(14) respectively. The conformation of the ten-membered ring is stabilized by the interactions between the C(9) carbonyl group and the conjugated groups of the planar D ring.

Fig. 1.  $C_{24}H_{32}O_6$ : perspective view of the molecule and torsion angles in the molecule.

(1'S,4'S,5'R)-1'-ETHYL-4'-(4'',8''-DIMETHYL-5''-OXONONYL)-4'-METHYL-3',8'-DIOXA-BICYCLO[3.2.1]OCTANE p-TOLUENESULFONYLHYDRAZONE

$C_{27}H_{44}N_2O_4S$

R.M. KANOJIA, M.P. WACHTER, S.D. LEVINE, R.E. ADAMS, R. CHEN, E. CHIN, M.L. COTTER, A.F. HIRSCH, R. HUETTEMANN, V.V. KANE, P. OSTROWSKI, C.J. SHAW, J.L. MATEOS, L. NORIEGA, A. GUZMAN, A. MIJAREZ, L. TOVAR and E. SHEFTER, 1982. J. Org. Chem., 47, 1310-1319.

Monoclinic,  $P2_1$ ,  $a = 5.378$ ,  $b = 18.973$ ,  $c = 14.182$  Å,  $\beta = 102.68^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.087$  for 1118 reflexions.

The analysis established the structure and absolute configuration as shown in Fig. 1.

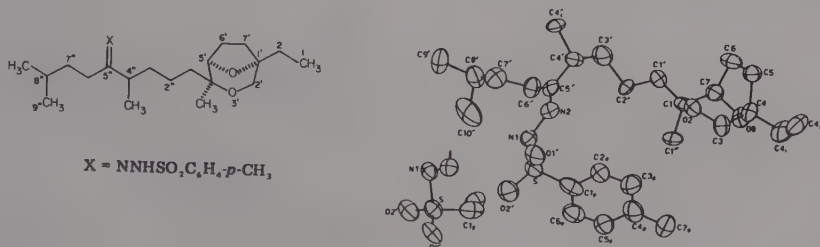


Fig. 1. The structure of  $C_{27}H_{44}N_2O_4S$ .

PSEUDOPTEROLIDE URETHANE MONOHYDRATE

$C_{28}H_{28}BrNO_7 \cdot H_2O$

M.M. BANDURRAGA, W. FENICAL, S.F. DONOVAN and J. CLARDY, 1982. J. Am. Chem. Soc., 104, 6463-6465.

Orthorhombic,  $P2_12_12$ ,  $a = 12.470$ ,  $b = 24.001$ ,  $c = 9.356$  Å,  $Z = 4$ . Cu radiation,  $R = 0.095$  for 2041 reflexions (absolute configuration).

The structure and absolute configuration of this irregular diterpenoid are shown in Fig. 1. This novel monocyclic skeleton is related in part to cubitene.

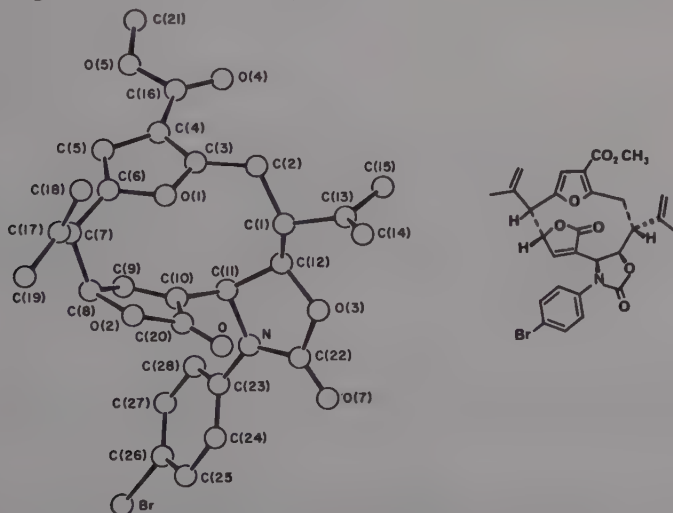


Fig. 1. The  $C_{28}H_{28}BrNO_7$  molecule. Stereochemical designators are: (1S), (7R), (8R), (11S), (12R).

## AJUGAREPTANSONE A



C. MIRAVITLLES, X. SOLANS, G. GERMAIN and J.P. DECLERCQ, 1982. *Acta Cryst.*, **B38**, 188-191.

Orthorhombic,  $P2_12_12_1$ ,  $a = 16.225$ ,  $b = 11.007$ ,  $c = 16.401$  Å,  $Z = 4$ . Cu radiation,  $R = 0.057$  for 3497 reflexions.

In the molecule (Fig. 1) the A, B rings adopt a trans-fused, chair-chair conformation. The absolute configuration, which was determined by the Bijvoet difference method, is the same as that of other clerodane compounds. Bond lengths and angles are generally as expected.

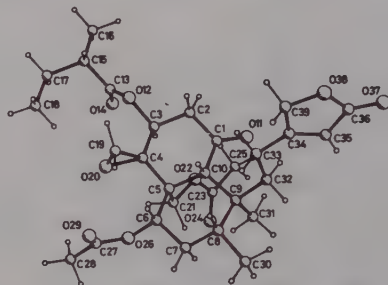
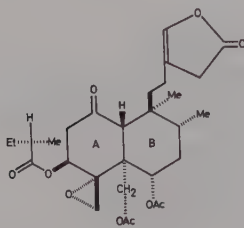
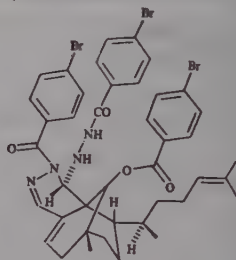
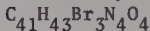


Fig. 1. A view of the  $C_{29}H_{40}O_{10}$  molecule.

(9R)-9-HYDROXYDICHOTOMA-2,14-DIENE-19,20-DIAL 4-BROMOBENZOATE BIS(4-BROMOBENZOIC ACID HYDRAZIDE)



J.F. BLOUNT, R.W. DUNLOP, K.L. ERICKSON and R.J. WELLS, 1982. *Aust. J. Chem.*, **35**, 145-163.

Triclinic,  $P1$ ,  $a = 16.211$ ,  $b = 16.366$ ,  $c = 17.637$  Å,  $\alpha = 63.63^\circ$ ,  $\beta = 76.68^\circ$ ,  $\gamma = 75.41^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.047$  for 8704 reflexions.

The structure shown above is confirmed with essentially the same conformation for all four molecules except for one phenyl ring which is rotated by different amounts. In the crystal the four molecules are joined into two pairs by hydrogen bonds ( $NH \cdots O$  2.92(1)-2.95(1) Å).

(1S,4aR,5S,6R,8aR)-1-HYDROXYMETHYL-5-{2-[(3R,6S)-3-(1R)-(2-HYDROXY-1-METHYLETHYL)-6-METHYL-1,2-DIOXAN-6-YL]ETHYL}-5,6,8a-TRIMETHYLBICYCLO[4.4.0]DECAN-1-OL



C. PICCINNI-LEOPARDI, G. GERMAIN, M. VAN MEERSSCHE, M. ALBERICCI, J.-C. BRAEKMAN, D. DALOZE and B. TURSCH, 1982. *J. Chem. Soc. Perkin II*, 1523-1526.

Orthorhombic,  $P2_12_12_1$ ,  $a = 6.272$ ,  $b = 14.094$ ,  $c = 26.818$  Å,  $Z = 4$ . Cu radiation,  $R = 0.047$  for 1632 reflexions.

The X-ray structure of this crystalline derivative (Fig. 1) of Sigmosceptrellin-B, from the sponge "Sigmosceptrella laevis", confirms the structure of the parent compound. The O(2)-O(3) bond length is 1.464(4) with O(3)-C(13) 1.435(5) and O(2)-C(16) 1.443(6) Å. The cyclic peroxide has a distorted chair conformation, with all the torsion angles involving the O atoms wider than the normal value of  $55^\circ$  in cyclohexane, (C(16)-O(2)-O(3)-C(13)  $72^\circ$ , C(15)-C(16)-O(2)-O(3)  $-65^\circ$ , C(14)-C(13)-O(3)-O(2)  $-64^\circ$ ). This ring has an approximate twofold axis passing through the mid-points of O(2)-O(3) and C(14)-C(15).

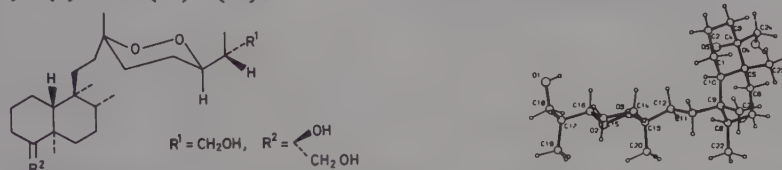
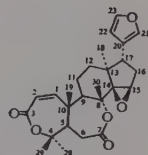


Fig. 1. Molecular skeleton and structure of  $\text{C}_{24}\text{H}_{44}\text{O}_5$ .

#### SURENOLACTONE

$\text{C}_{26}\text{H}_{32}\text{O}_6$



W. KRAUS, K. KYPKE, M. BOKEL, W. GRIMMINGER, G. SAWITZKI and G. SCHWINGER, 1982. Justus Liebigs Ann. Chem., 87-98.

Orthorhombic,  $P2_12_12_1$ ,  $a = 8.200$ ,  $b = 9.752$ ,  $c = 28.978$  Å,  $Z = 4$ . Mo radiation,  $R = 0.048$  for 1790 reflexions (at  $-100^\circ\text{C}$ ).

The structure was established as that shown above with normal values for bond lengths and angles.

#### ABRUSLACTONE A

$\text{C}_{30}\text{H}_{46}\text{O}_3$

H.M. CHANG, T.C. CHIANG and T.C.W. MAK, 1982. J. Chem. Soc. Chem. Commun., 1197-1198.

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.321$ ,  $b = 12.820$ ,  $c = 26.864$  Å,  $D_m = 1.12$ ,  $Z = 4$ . Mo radiation,  $R = 0.063$  for 1564 reflexions.

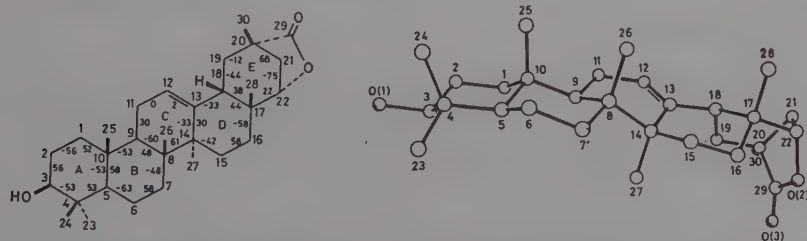


Fig. 1.  $\text{C}_{30}\text{H}_{46}\text{O}_3$ : the molecular skeleton showing selected torsion angles and a perspective view of the molecule.

The molecule, a new triterpenoid sapogenin isolated from the roots and vines of 'Abrus precatorius L.' has the  $\gamma$ -lactone of  $3\beta,22\alpha$ -dihydroxyolean-12-en-29 $\alpha$ -oic acid with ring E in a boat conformation (Fig. 1). Rings A, B and D have chair conformations and ring C a twist conformation due to the presence of an ethylenic double bond. The molecular dimensions are normal. Molecules related by the  $2_1$  screw axis parallel to 'a' are linked by O(1)-H...O(3') hydrogen bonds (2.872 Å) to form infinite zigzag chains in the crystal lattice.

### $3\beta$ -METHOXY-22 $\alpha$ -METHYL-NORSERRAT-14-EN-21-ONE



T.P. HAROMY and M. SUNDARALINGAM, 1982. Acta Cryst., B38, 1365-1367.

Orthorhombic,  $P2_12_12_1$ ,  $a = 9.598$ ,  $b = 10.559$ ,  $c = 25.150$  Å,  $D_m = 1.15$ ,  $Z = 4$ . Cu radiation,  $R = 0.037$  for 2373 reflexions.

In the molecule (Fig. 1) the A, B and E rings are in chair conformations, the seven-membered C ring is in a twist-chair conformation, and the six-membered D ring is in a half-chair conformation.

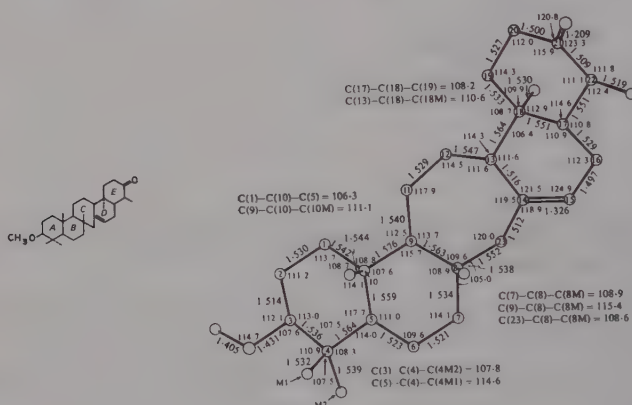
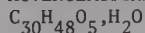


Fig. 1.  $C_{30}H_{48}O_2$ : bond distances and angles.

### HOVENOLACTONE MONOHYDRATE



Y. KOBAYASHI, T. TAKEDA, Y. OGIHARA and Y. IITAKA, 1982. J. Chem. Soc. Perkin I, 2795-2799.

Orthorhombic,  $P2_12_12_1$ ,  $a = 14.474$ ,  $b = 28.584$ ,  $c = 6.749$  Å,  $Z = 4$ . Cu radiation,  $R = 0.08$  for 2132 reflexions.

The structure and relative stereochemistry of this novel triterpene from 'Hovenia dulcis' (Fig. 1) are established by the X-ray structure. The bond lengths in the spiro lactone ring are C(14)-C(15) 1.546, C(15)-C(16) 1.497, C(16)-O(2) 1.322, O(2)-C(30) 1.430 and C(30)-C(14) 1.572 Å, and those in the epoxide are C(17)-C(20) 1.488, C(20)-O(4) 1.469 and C(17)-O(4) 1.456 Å.

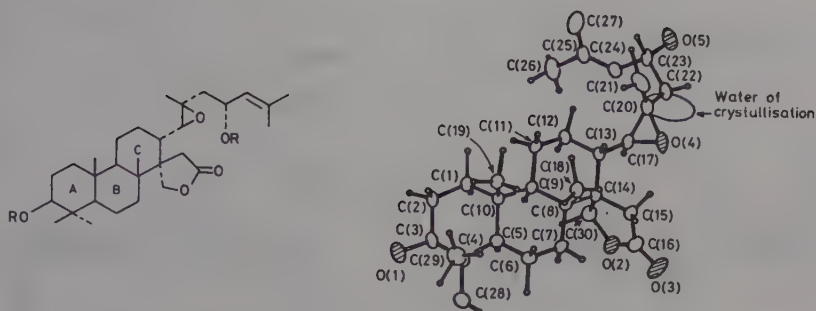


Fig. 1. Molecular skeleton ( $R = H$ ) and structure of  $C_{30}H_{48}O_5 \cdot H_2O$ .

21-ISOPROPYL-4 $\beta$ ,5 $\beta$ ,9 $\beta$ ,13 $\alpha$ ,14 $\beta$ ,17 $\alpha$ -HEXAMETHYLPERHYDROCYCLOPENTA[a]CHRYSEN-3-ONE  
(3-FILICANONE)

$C_{30}H_{50}O$

B.F. PEDERSEN, 1982. *Acta Cryst.*, B38, 1652-1654.

Monoclinic,  $P2_1$ ,  $a = 13.538$ ,  $b = 6.342$ ,  $c = 14.797$  Å,  $\beta = 96.04^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.09$  for 1703 reflexions.

The molecule (Fig. 1) has an extended structure with all rings trans fused. The six-membered rings have deformed chair conformations while the five-membered ring has a twisted envelope conformation with C(17) 0.86(2) Å out of the plane. The methyl groups at positions 5,9,13,14 and 17 are axial while that at position 4 is equatorial. Non-bonded interactions between methyl groups result in some bending and twisting along the major axis of the molecule. Bond lengths and angles are normal.

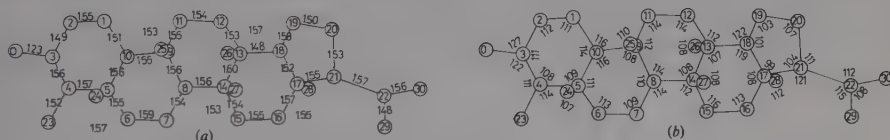


Fig. 1. The 3-filicanone molecule with (a) bond lengths (b) bond angles.

STICTANE-3 $\beta$ ,22 $\alpha$ -DIOL

$C_{30}H_{52}O_2$  (I)

22 $\alpha$ -HYDROXY-3,4-SECOSTICTAN-3-OIC ACID ACETONE SOLVATE

$C_{30}H_{52}O_3 \cdot C_2H_4O$  (II)

R.E. CORBETT, J. SIMPSON, E.M. GOH, B.K. NICHOLSON, A.L. WILKINS and W.T. ROBINSON, 1982. *J. Chem. Soc. Perkin II*, 1339-1343.

I. Monoclinic,  $C2$ ,  $a = 24.14$ ,  $b = 7.606$ ,  $c = 15.900$  Å,  $\beta = 117.3^\circ$ ,  $D_m = 1.13$ ,  $Z = 4$ . Cu radiation,  $R = 0.057$  for 1793 reflexions.

II. Monoclinic,  $P2_1$ ,  $a = 8.938$ ,  $b = 27.789$ ,  $c = 6.687$  Å,  $\beta = 111.36^\circ$ ,  $D_m = 1.12$ ,  $Z = 2$ . Cu radiation,  $R = 0.087$  for 887 reflexions.

The X-ray study confirms the structure of these triterpenes (Fig. 1) from "Pseudocyphellaria" lichen species. Ring B in I has a twisted boat conformation. Steric crowding around C(8), C(9) and C(10) results in lengthening of the C(8)-C(14) (1.614) and C(9)-C(10) (1.588) bonds, while C(5)-C(6) (1.509 Å) is shortened. The corresponding B ring in II has a chair conformation and unremarkable bond lengths within the precision of the results.



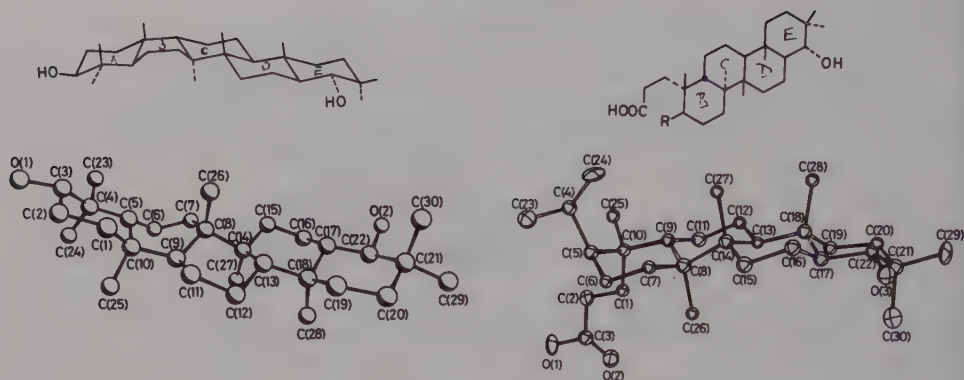
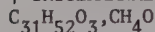


Fig. 1. Molecular skeletons and structures of  $C_{30}H_{52}O_2$  (upper) and  $C_{30}H_{52}O_3, C_2H_6O$  ( $R = CH(CH_3)_2$ ) (lower).

$\gamma$ -IRIGERMANAL METHANOL SOLVATE



F.-J. MARNER, W. KRICK, B. GELLRICH, L. JAENICKE and W. WINTER, 1982. J. Org. Chem., 47, 2531-2536.

Monoclinic,  $P2_1$ ,  $a = 13.05$ ,  $b = 8.84$ ,  $c = 13.78 \text{ \AA}$ ,  $\beta = 105.84^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.114$  for 1842 reflexions.

All bond distances and angles in the molecule (Fig. 1) are in the expected range, with exception of the cyclohexane bonds  $C(10)-C(6)$ ,  $C(10)-C(11)$ , and  $C(23)-C(18)$ ,  $C(23)-C(22)$ , which seem to be significantly stretched to 1.57-1.59(2)  $\text{\AA}$  by crowding with four nonhydrogen substituents at  $C(11)$  and  $C(22)$ . Molecules are linked by hydrogen bonds.

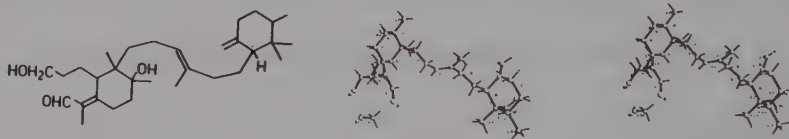


Fig. 1. The  $\gamma$ -irigermanal molecule.

BAURENYL ACETATE



B. TINANT, G. GERMAIN, J.P. DECLERCQ, M. VAN MEERSSCHE, J.F. CICCIO and P. HOET, 1982. Bull. Soc. Chim. Belg., 91, 117-121.

Orthorhombic,  $P2_12_12_1$ ,  $a = 8.291$ ,  $b = 11.084$ ,  $c = 30.041 \text{ \AA}$ ,  $Z = 4$ . Mo radiation,  $R = 0.064$  for 975 reflexions.

The C-C single-bond lengths in the triterpenoid nucleus (Fig. 1) range between 1.498 and 1.582  $\text{\AA}$  with a mean value of 1.545  $\text{\AA}$ . The deviations from the mean are not exceptional in comparison with other triterpenoid structures and are probably caused by strain in the molecule. The distances of the double bond  $C(7)=C(8)$  is 1.338  $\text{\AA}$ . The conformations of the five six-membered rings in the triterpenoid nucleus are chair for ring A, boat for ring B, skew for ring C, chair for ring D and boat for ring E. The  $3\beta$ -acetate group is in equatorial position and the carbonyl group  $C(31)=O(33)$

is synperiplanar to C(3), the torsion angle C(3)-O(3)-C(31)-O(33) being  $-3^\circ$ .

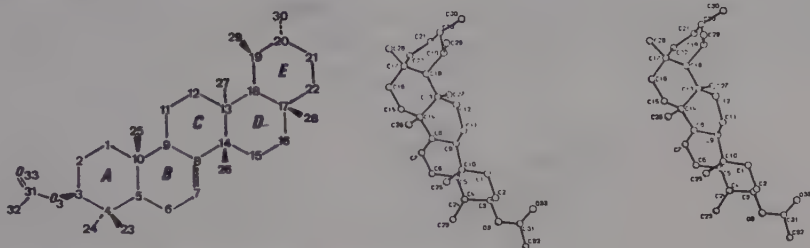
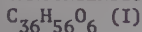
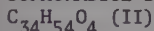


Fig. 1. Baurenyl acetate and a stereodrawing.

#### CANTONIENSISTRIOL TRIACETATE



#### SOPHORADIOL DIACETATE



T.C.W. MAK, T.-C. CHIANG and H.-M. CHANG, 1982. J. Chem. Soc. Chem. Commun., 785-786.

I. Monoclinic,  $P2_1$ ,  $a = 12.238$ ,  $b = 7.356$ ,  $c = 18.804 \text{ \AA}$ ,  $\beta = 91.78^\circ$ ,  $D_m = 1.12$ ,  $Z = 2$ . Mo radiation,  $R = 0.109$  for 2057 reflexions.

II. Orthorhombic,  $P2_12_12_1$ ,  $a = 7.407$ ,  $b = 13.180$ ,  $c = 30.919 \text{ \AA}$ ,  $D_m = 1.16$ ,  $Z = 4$ . Cu radiation,  $R = 0.095$  for 1436 reflexions.

I is a new triterpenoid sapogenol isolated from the roots of the medicinal Chinese herb 'Abrus cantoniensis Hance' and is shown (Fig. 1) to be an olean-12-ene- $3\beta,21\beta,22\beta$ -triol; II, from the same source is an olean-12-ene- $3\beta,22\beta$ -diol. In both molecules the A/B and B/C ring junctions are trans-fused and the D/E ring junction cis-fused. Ring C has a twist conformation on account of the double bond. The molecular dimensions are normal for this type of compound.

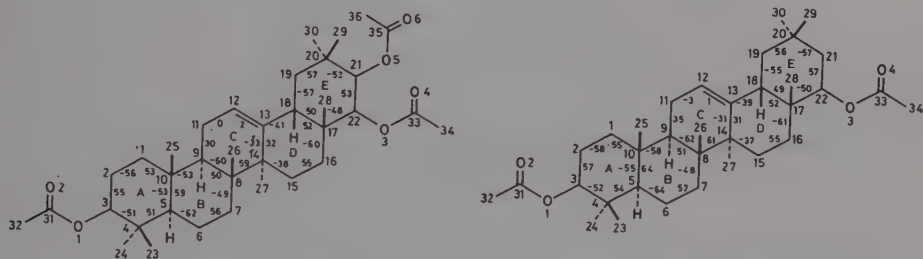
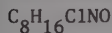


Fig. 1. Atom numbering and torsion angles in  $\text{C}_{36}\text{H}_{56}\text{O}_6$  (left) and  $\text{C}_{34}\text{H}_{54}\text{O}_4$  (right).

#### PHYSOPERUVINE HYDROCHLORIDE



A.B. RAY, 1982. Heterocycles, 19, 1233-1236.

Orthorhombic,  $P2_12_12_1$ ,  $a = 10.282$ ,  $b = 10.026$ ,  $c = 8.900 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.086$  for 872 reflexions.

The absolute configuration of "physoperuvine" is that shown in Fig. 1, and the hydrochloride of physoperuvine exists exclusively in the amino-alcohol form. The  $\text{Cl}^-$  ion is involved in N-H...Cl and O-H...Cl hydrogen bonds with adjacent molecules,

forming a network in the *c* axis direction. The six-membered ring has a chair-, and the seven-membered ring a boat conformation. Bond distances are: C(1)-C(2) 1.567, C(2)-C(3) 1.566, C(3)-C(4) 1.569, C(4)-C(5) 1.512, C(5)-C(6) 1.541, C(6)-C(7) 1.564, C(7)-C(1) 1.558, C(1)-N 1.567, C(5)-N 1.536, N-C(8) 1.513, N...Cl 3.08, N-H...Cl 2.05, O...Cl 3.04, N-H...Cl 2.08 Å.

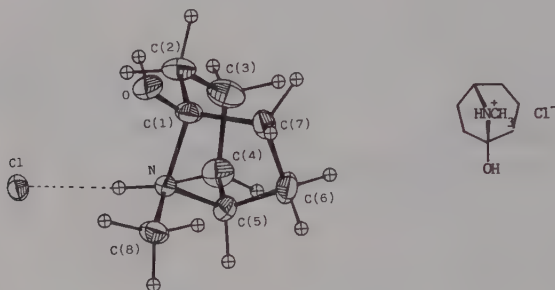


Fig. 1. Molecular structure of C<sub>8</sub>H<sub>16</sub>ClNO and line diagram of amino-alcohol form.

#### HARMINE (7-METHYL-9H-PYRIDO[3,4-*b*]INDOLE)

C<sub>13</sub>H<sub>12</sub>N<sub>2</sub>O

P.R. MAULIK and B.S. BASAK, 1982. Cryst. Struct. Comm., **11**, 19-24.

Orthorhombic, P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 9.56, *b* = 18.95, *c* = 5.811 Å, D<sub>m</sub> = 1.35, Z = 4. Cu radiation, R = 0.103, for 1169 reflexions.

A view of the molecule with dimensions is in Fig. 1. Molecules are linked by N-H...N hydrogen bonds (N...N 2.979(3) Å).

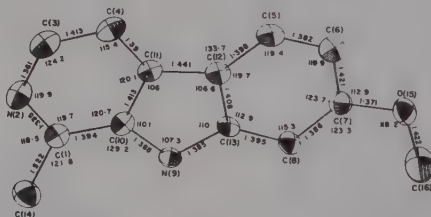


Fig. 1. The harmine molecule with dimensions (e.s.d.'s are 0.009-0.014 Å, 0.6-0.9°).

#### VIRGIDIVARINE

C<sub>15</sub>H<sub>26</sub>N<sub>2</sub>O<sub>2</sub>

A.J. DE KOK, C. ROMERS and J.L. VAN EIJK, 1982. Acta Cryst., **B38**, 466-468.

Tetragonal, P4<sub>1</sub>2<sub>1</sub>2 or P4<sub>3</sub>2<sub>1</sub>2, *a* = 8.339, *c* = 43.537 Å, Z = 8. Mo radiation, R = 0.043 for 875 reflexions.

The molecule (Fig. 1) is a zwitterion with N(1') involved in two intermolecular hydrogen bonds, N(1')-H...O(12) and N(1')-H...O(13) of length 2.658(5) and 2.740(5) Å respectively. The piperidine rings have chair conformations with average puckering angles 57.1 and 56.6°.

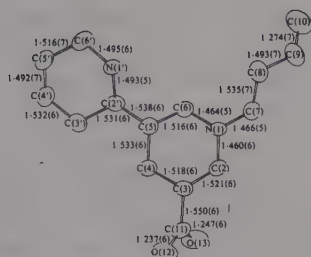


Fig. 1. The virgidivarine molecule.

1-(2'-AMINOPYRIMIDIN-4'-YL)-6-METHOXY- $\beta$ -CARBOLINE ETHANOL SOLVATE (METHOXYANNOMONTINE ETHANOL SOLVATE)

$C_{16}H_{13}N_5O, C_2H_6O$

M. LEBOEUF, A. CAVE, P. FORGACS, J. PROVOST, A. CHIARONI and C. RICHE, 1982. J. Chem. Soc. Perkin I, 1205-1208.

Triclinic,  $P\bar{1}$ ,  $a = 13.207$ ,  $b = 8.831$ ,  $c = 8.200$  Å,  $\alpha = 83.0$ ,  $\beta = 110.0$ ,  $\gamma = 110.0^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.108$  for 1513 reflexions.

This novel alkaloid (Fig. 1) is approximately planar, the angle between the  $\beta$ -carboline and pyrimidine mean planes being  $4^\circ$ . An intramolecular hydrogen bond length of 2.74 Å is observed between N(9) and N(1'). The solvating ethanol bridges two molecules of methoxyannomontine, is disordered and can occupy two positions in which the hydroxyl groups are superimposed. The molecular dimensions are imprecise.

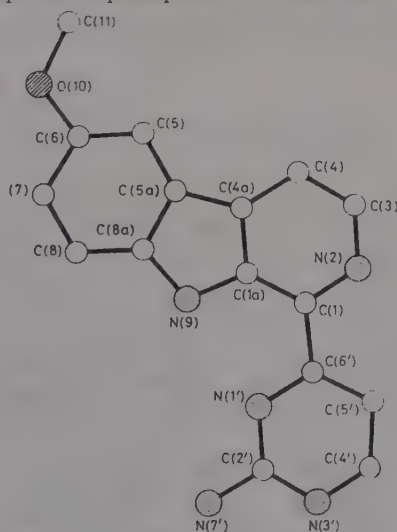


Fig. 1. Methoxyannomontine.

4,5 $\alpha$ -EPOXY-3,14-DIHYDROXY-17-METHYLMORPHINAN-6-ONE HYDROCHLORIDE MONOHYDRATE ETHANOL SOLVATE

$C_{17}H_{20}ClNO_4, H_2O, C_2H_6O$

$C_{17}H_{20}NO_4^+, Cl^-, H_2O, C_2H_5OH$

S.D. DARLING, V.M. KOLB, G.S. MANDEL and N.S. MANDEL, 1982. *J. Pharm. Sci.*, **71**, 763-767.

Orthorhombic,  $P2_12_12_1$ ,  $a = 10.586$ ,  $b = 18.671$ ,  $c = 9.361$  Å,  $D_m = 1.343$ ,  $Z = 4$ . Mo radiation,  $R = 0.056$  for 1710 reflexions.

The structure analysis was undertaken to establish whether protonation of N atoms influences the geometry of morphines. There are some significant differences in bond lengths of protonated and unprotonated (1) oxymorphone. The bond N-C(9) is longer by 0.032 Å in the protonated form and several C-C bonds are shorter (by 0.036-0.054 Å) - namely bonds C(1)-C(2), C(10)-C(11), C(5)-C(13), C(9)-C(14), C(11)-C(12) and C(4)-C(12) (Fig. 1).

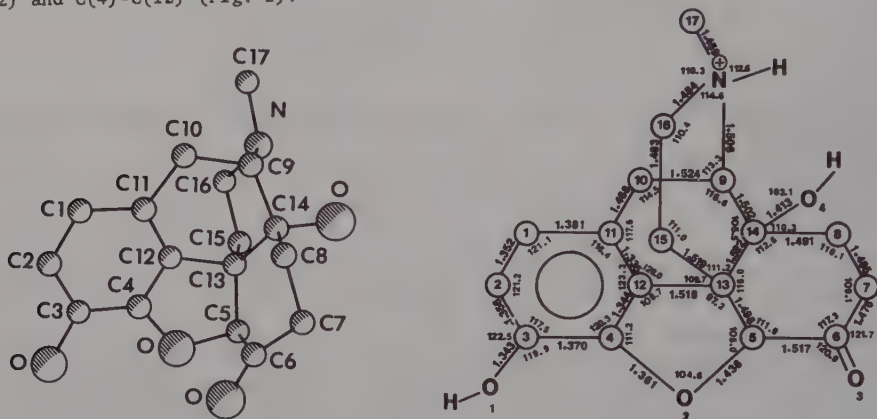
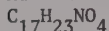


Fig. 1. Perspective drawing of  $C_{17}H_{20}NO_4^+$  and bond lengths and angles.

1. Structure Reports, **42B**, 452.

#### HIPPEASTIDINE



W.H. WATSON, V. ZABEL, M. SILVA and P. PACHECO, 1982. *Cryst. Struct. Comm.*, **11**, 157-162.

Orthorhombic,  $P2_12_12_1$ ,  $a = 11.831$ ,  $b = 12.881$ ,  $c = 10.143$  Å,  $Z = 4$ . Cu radiation,  $R = 0.051$  for 1533 reflexions.

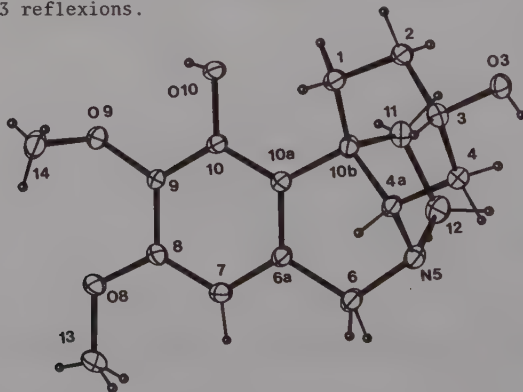
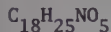


Fig. 1. The alkaloid hippeastidine.

In this alkaloid (Fig. 1) the six-membered ring 1-2-3-4-4a-10b exhibits a chair conformation while ring 4a-N(5)-6-6a-10a-10b is described as 1,2-diplanar. The five-membered ring is a distorted envelope. The proton of O(10) is hydrogen bonded to O(3) (O(3)...O(10) 2.757(5) Å), while the proton of O(3) is hydrogen bonded to N(5) (O(3)...N(5) 2.849(5) Å).

## SENECIONINE



M.F. MACKAY and C.C.J. CULVENOR, 1982. *Acta Cryst.*, **B38**, 2754-2758.

Orthorhombic,  $P2_12_12_1$ ,  $a = 9.072$ ,  $b = 17.236$ ,  $c = 11.224$  Å,  $D_m = 1.266$ ,  $Z = 4$ . Cu radiation,  $R = 0.046$  for 1694 reflexions.

In the molecule (Fig. 1) the pyrrolizidine ring is exo-buckled at an angle of  $35.3(4)^\circ$  and the ester carbonyl bonds are anti-parallel. Bond lengths and angles agree well with those in other pyrrolizidine alkaloids. Intermolecular hydrogen bonds involving the hydroxyl substituent and the N atoms,  $N...O$  2.827(4) Å, link the molecules in the crystal into helices extending along the  $c$  axis.

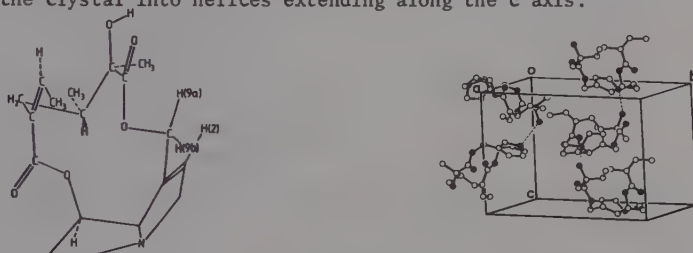


Fig. 1.  $\text{C}_{18}\text{H}_{25}\text{NO}_5$ : structural formula and molecular packing in the crystal; hydrogen bonds are indicated by dashed lines.

(-)-(1aR,6bR,6aS,10R,11R)-10-HYDROXY-13-cis-ETHYLIDENE-10,11-DIMETHYL-1a,2,5,6,6a,6b-HEXAHYDRO-3H-PYRROLIZINO[1a,6b,6a-b,c]-1,8-DIOXADUODECANE-9,14-DIONE  
 $\text{C}_{18}\text{H}_{27}\text{NO}_5$

H. WIEDENFELD, E. RÖDER, A. KIRFEL and G. WILL, 1982. *Arch. Pharm.*, **315**, 165-169.

Orthorhombic,  $P2_12_12_1$ ,  $a = 11.405$ ,  $b = 17.121$ ,  $c = 9.194$  Å,  $Z = 4$ . Mo radiation,  $R = 0.086$  for 821 reflexions.

The two five-membered rings show the expected half-chair conformation with C(6) below, and C(2) above the projection plane (Fig. 1). Bond distances of interest are: C(8)-N 1.561, C(1)-C(9) 1.532, C(9)-O(1) 1.474, O(1)-C(10) 1.351, C(10)-O(2) 1.169, C(10)-C(11) 1.551, C(11)-O(3) 1.425, C(11)-C(12) 1.577, C(12)-C(13) 1.571, C(13)-C(14) 1.569, C(14)-C(18) 1.350, C(14)-C(15) 1.496, C(15)-O(5) 1.207, C(15)-O(4) 1.357, O(4)-C(7) 1.485 Å.

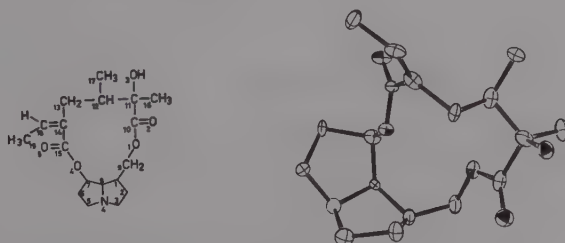


Fig. 1. Atomic labelling scheme, and absolute configuration of  $\text{C}_{18}\text{H}_{27}\text{NO}_5$ .



JUNCEINE  
 $C_{18}H_{27}NO_7$

H. STOECKLI-EVANS, 1982. *Acta Cryst.*, **B38**, 1614-1617.

Orthorhombic,  $C222_1$ ,  $a = 12.285$ ,  $b = 16.861$ ,  $c = 17.502$  Å,  $D_m = 1.34$ ,  $Z = 8$ . Mo radiation,  $R = 0.050$  for 1011 reflexions.

The 11-membered macrocycle has the ester carbonyls synparallel and directed below the plane of the ring (Fig. 1). The pyrrolizidine ring is in the exo-puckered form; the interplanar angles  $C(5)C(6)C(7)/C(5)N(4)C(8)$  and  $C(1)C(2)C(3)N(4)C(8)/C(5)N(4)C(8)C(7)$  are  $36.7(6)$  and  $125.7(5)^\circ$ . The  $N(4)-C(8)$  distance is  $1.500(8)$  Å. There are two intramolecular hydrogen bonds;  $O(22)\dots O(19)$   $2.61(1)$  and  $O(19)\dots O(21)$   $2.67(1)$  Å and two intermolecular ones;  $O(21)\dots N(4)$   $2.77(1)$  and  $O(22)\dots O(17)$   $3.02(1)$  Å linking molecules in the  $b$  and  $a$  directions. The shortest transannular distance is  $C(11)\dots O(16)$  at  $2.92(1)$  Å.

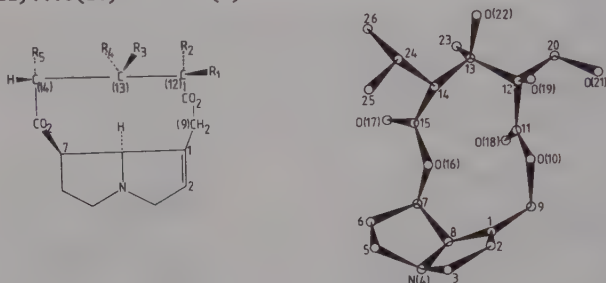


Fig. 1. The junceine molecule ( $R_1 = R_3 = OH$ ,  $R_2 = CH_2OH$ ,  $R_4 = CH_3$ ,  $R_5 = CH(CH_3)_2$ ).

TRIMETHYLCOLCHICINIC ACID HYDROCHLORIDE HYDRATE  
 $C_{19}H_{22}ClNO_5 \cdot 1.75(H_2O)$

T.N. MARGULIS, 1982. *Cryst. Struct. Comm.*, **11**, 1689-1694.

Monoclinic,  $C2$ ,  $a = 13.449$ ,  $b = 8.295$ ,  $c = 36.969$  Å,  $\beta = 101.38^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.048$  for 3407 reflexions.

Both independent cations (Fig. 1) are in the colchicine rather than isocolchicine form. The troponoid ring C has alternating single and double bonds as well as non-zero torsion angles ( $-20$  to  $+16^\circ$ ). Hydrogen bonds link cations to water, chloride ions, and other cations.

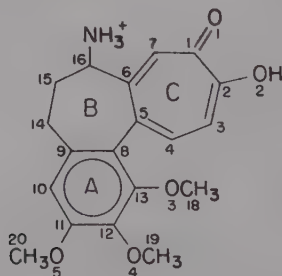
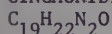


Fig. 1. The cation in  $C_{19}H_{22}ClNO_5 \cdot 1.75(H_2O)$ .

## CINCHONIDINE



B.J. OLEKSYN, 1982. Acta Cryst., B38, 1832-1834.

Orthorhombic,  $P2_12_12_1$ ,  $a = 10.941$ ,  $b = 20.883$ ,  $c = 7.130$  Å,  $D_m = 1.22$ ,  $Z = 4$ . Mo radiation,  $R = 0.05$  for 1626 reflexions.

The molecular structure is shown in Figure 1. Molecular dimensions, conformation and packing are similar to those in the RS optical isomer cinchonine (1). Molecules are hydrogen-bonded into chains. There are no intramolecular hydrogen bonds.

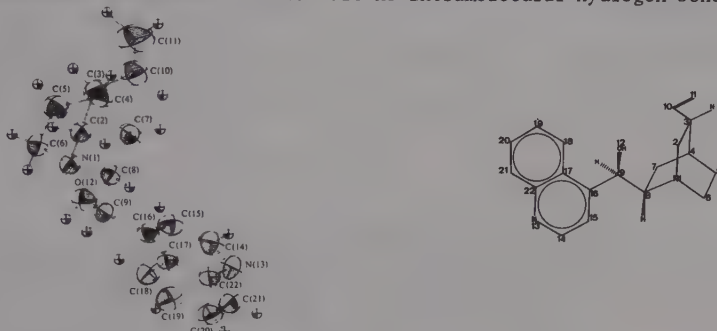
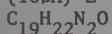


Fig. 1. The cinchonidine molecule.

1. Structure Reports, 45B, 637.

(168H)- $\Delta^{14}$ -VINCANOL

N. RODIER, S. BAASSOU, H. MEHRI and M. PLAT, 1982. Acta Cryst., B38, 863-867.

Orthorhombic,  $P2_12_12_1$ ,  $a = 10.285$ ,  $b = 16.30$ ,  $c = 18.38$  Å,  $Z = 8$ . Mo radiation,  $R = 0.047$  for 2497 reflexions.

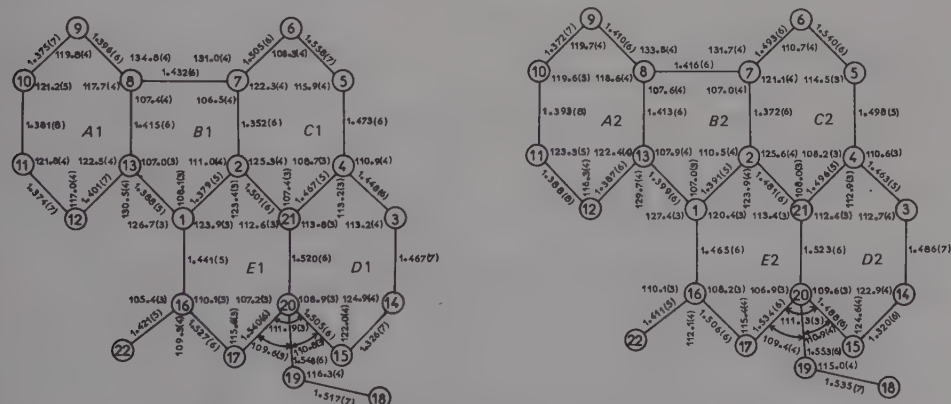


Fig. 1.  $\text{C}_{19}\text{H}_{22}\text{N}_2\text{O}$ : bond distances and angles in the two independent molecules.

The asymmetric unit contains two independent molecules (Fig. 1). The ring conformations are sofa for C and E1, half-chair for D and chair for E2. The ring-junction configurations are planar for A/B, B/C and B1/E1, cis for C/D and D/E,

trans for C/E and quasi-trans for B2/E2. The structure consists of layers which run parallel with the (001) plane and its cohesion is due to hydrogen bonds and van der Waals interactions.

(-)-(1R,5R,9R,13R)-2'-HYDROXY-5,9 $\alpha$ -DIMETHYL-2-(TETRAHYDROFURFURYL)-6,7-BENZOMORPHAN  
C<sub>19</sub>H<sub>27</sub>NO<sub>2</sub> (I)

(-)-(1R,5R,9R,13S)-2'-HYDROXY-5,9-DIMETHYL-2-(TETRAHYDROFURFURYL)-6,7-BENZOMORPHAN  
TARTRATE MONOHYDRATE  
C<sub>23</sub>H<sub>33</sub>NO<sub>8</sub>·H<sub>2</sub>O (II)

O.M. PEETERS, C.J. DE RANTER and N.M. BLATON, 1982. Acta Cryst., B38, 3055-3059.

I. Orthorhombic, P<sub>2</sub><sub>1</sub>2<sub>1</sub>2<sub>1</sub>, a = 9.0022, b = 12.2825, c = 14.840 Å, D<sub>m</sub> = 1.23, Z = 4. Cu radiation, R = 0.046 for 1524 reflexions.

II. Monoclinic, P2<sub>1</sub>, a = 9.033, b = 16.106, c = 7.821 Å,  $\beta$  = 92.58°, D<sub>m</sub> = 1.38, Z = 2. Mo radiation, R = 0.038 for 1689 reflexions.

In both compounds (Fig. 1) the aromatic ring is almost planar, the hydroaromatic ring has sofa conformation and the piperidine ring is in slightly distorted chair conformation. In (I) molecules are linked by OH...N hydrogen bonds; in (II) there is an extensive hydrogen bond network involving the water and tartrate moieties.

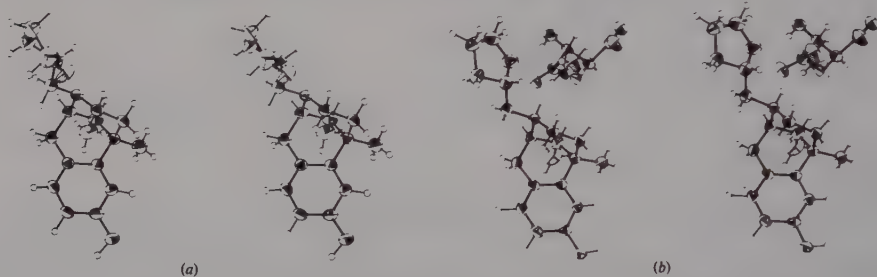


Fig. 1. Stereoviews of the benzomorphan derivatives (I) (a), and (II) (b).

#### O-METHYLFLAVINANTINE HYDRATE

C<sub>20</sub>H<sub>23</sub>NO<sub>4</sub>·H<sub>2</sub>O

A. DUBOURG, P. BRIARD, R. ROQUES, J.P. DECLERCQ and G. GERMAIN, 1982. Acta Cryst., B38, 1657-1659.

Orthorhombic, P<sub>2</sub><sub>1</sub>2<sub>1</sub>2<sub>1</sub>, a = 10.391, b = 12.456, c = 14.420 Å, Z = 4. Cu radiation, R = 0.10 for 1182 reflexions.

The molecule is as shown in Fig. 1. Ring B has a chair and ring C an envelope conformation. Molecules are linked by hydrogen bonds to the water molecule.

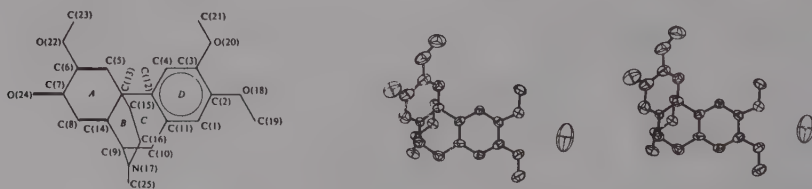
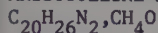


Fig. 1. The O-methylflavinantine molecule and a stereoview.

## ARISTOTELINE METHANOL ADDUCT



W.H. WATSON, V. ZABEL, M. SILVA and M. BITTNER, 1982. *Cryst. Struct. Comm.*, **11**, 141-146.

Orthorhombic,  $P2_12_12_1$ ,  $a = 13.158$ ,  $b = 21.816$ ,  $c = 6.563$  Å,  $Z = 4$ . Cu radiation,  $R = 0.051$  for 1806 reflexions.

In this alkaloid (Fig. 1) the indole moiety is planar as indicated by the torsion angles and there is considerable delocalization in the five-membered ring. The two C-N(6) distances and the C(10a)-C(10b) distance are reduced to 1.373(3), 1.376(3) and 1.442(3) Å while C(5a)-C(10b) is lengthened to 1.363(3) Å. The two six-membered rings N(1)-2-3-4-4a-11a and 3-4-5a-5-12-13 exhibit chair conformations while 4a-5-5a-10b-11-11a is in a half-chair conformation. The two nitrogen atoms are involved in hydrogen bonding with the methanol solvate molecule. N(6) acts as a proton donor, N(6)...O(18) 2.849(4) Å, and N(1) acts as a proton acceptor, N(1)...O(18) 2.801(3) Å.

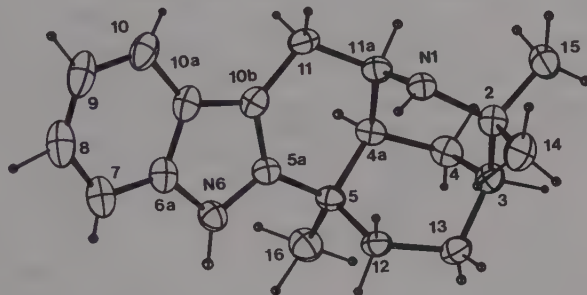
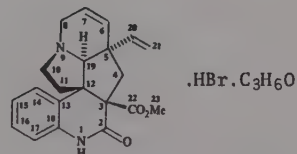
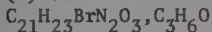


Fig. 1. A drawing of aristoteline.

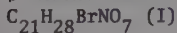
## (+)-SCANDINE HYDROBROMIDE ACETONE SOLVATE



J.R. CANNON, K.D. CROFT, Y. MATSUKI, V.A. PATRICK, R.F. TOIA and A.H. WHITE, 1982. *Aust. J. Chem.*, **35**, 1655-1664.

Monoclinic,  $P2_1$ ,  $a = 9.496$ ,  $b = 14.561$ ,  $c = 9.339$  Å,  $\beta = 115.39^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.037$  for 2657 reflexions.

The absolute configuration of (+)-scandine was confirmed as that shown above. The segment C(13)C(18)N(1)C(2) is virtually planar; N(1) is conjugated primarily with the carbonyl as an amide with little conjugation to the aromatic ring (N(1)-C(2) 1.343(6), N(1)-C(18) 1.396(8), C(2)-O(2) 1.221(8) Å). A number of bonds at the ring junctions are lengthened significantly (C(3)-C(4), C(12) 1.560(7), 1.560(9), C(5)-C(19) 1.578(7), C(12)-C(11), C(19) 1.578(7), 1.586(8), N(9)-C(8), C(10), C(19) 1.506(7), 1.531(8), 1.521(6) Å). The C(20)-C(21) is unusually short (1.259(11) Å).

 $\beta$ -HYDROXYLAUDONOSINE HYDROBROMIDE

## 7-HYDROXYGLAUCINE METHIODIDE METHANOL SOLVATE

 $C_{22}H_{28}INO_5 \cdot CH_4O$  (II)

## 13-HYDROXYXYLOPININE METHIODIDE HEMIHYDRATE METHANOL SOLVATE

 $C_{22}H_{28}INO_5 \cdot 0.5(CH_4O) \cdot 0.5(H_2O)$  (III)

K.L. WERT, S. CHACKALAMANNIL, E. MILLER, D.R. DALTON, D.E. ZACHARIAS and J.P. GLUSKER, 1982. *J. Org. Chem.*, **47**, 5141-5150.

I. Monoclinic,  $Cc$ ,  $a = 10.727$ ,  $b = 27.901$ ,  $c = 7.152 \text{ \AA}$ ,  $\beta = 97.52^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.039$  for 1964 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 11.085$ ,  $b = 19.835$ ,  $c = 10.795 \text{ \AA}$ ,  $\alpha = 94.49^\circ$ ,  $\beta = 96.52^\circ$ ,  $\gamma = 83.66^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.087$  for 7541 reflexions.

III. Triclinic,  $P\bar{1}$ ,  $a = 11.668$ ,  $b = 20.644$ ,  $c = 10.512 \text{ \AA}$ ,  $\alpha = 97.94^\circ$ ,  $\beta = 109.28^\circ$ ,  $\gamma = 75.90^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.087$  for 6350 reflexions.

The structures of the three alkaloid cations are shown in Fig. 1. In I, the torsion angle  $N-C1-C13-O$  is  $-64^\circ$  and is related, in part, to the  $N-H \cdots Br$  and  $O-H \cdots Br$  hydrogen bonds which form in the crystalline state. In II, the  $N-C6-C7-O$  and  $N-C6-C7-H7$  torsion angles are sterically fixed at the nonanticoplanar values  $73^\circ$  and  $-47^\circ$ , respectively. The  $H6-C6-C7-O$  torsion angle is  $-167^\circ$ , approaching anti-coplanarity, and promotes dehydration to form the  $C6-C7$  double bond. In III, the  $N-C14-C13-O$  torsion angle is sterically fixed at  $155^\circ$ ,  $25^\circ$  away from anticoplanarity and unfavorable for Hofmann elimination with participation of oxygen or hydrogen to give either epoxide or ketone product.

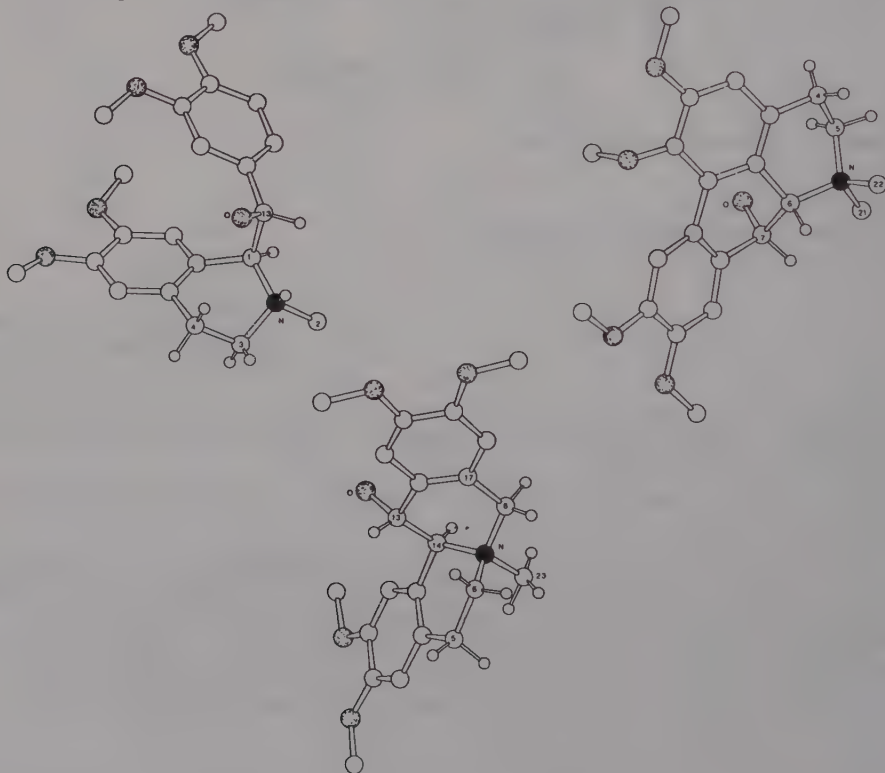


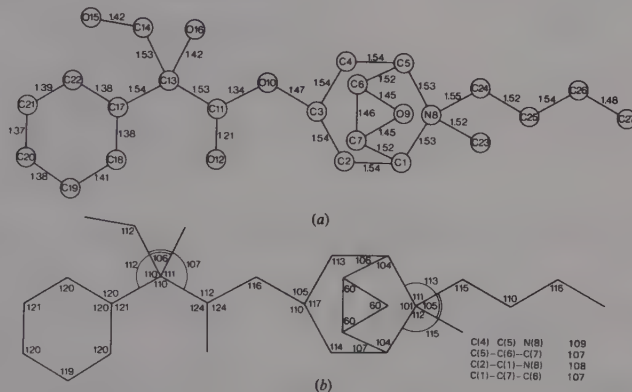
Fig. 1. The cations of  $\beta$ -hydroxyglauconosine (I) (top left), 7-hydroxyglauconosine (II) (top right), and 13-hydroxyxylopinine (III) (bottom).

6 $\beta$ ,7 $\beta$ -EPOXY-1 $\alpha$ H,5 $\alpha$ H-TROPAN-3 $\alpha$ -YL(-)-2,3-DIHYDROXY-2-PHENYLPROPIONATE n-BUTYL BROMIDE  
 $C_{21}H_{30}BrNO_5$   $C_{17}H_{21}NO_5, C_4H_9Br$

J. BODE and C.H. STAM, 1982. Acta Cryst., B38, 333-335.

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.215$ ,  $b = 12.974$ ,  $c = 22.466$  Å,  $Z = 4$ . Cu radiation,  $R = 0.027$  for 1978 reflexions.

Bond distances and angles (Fig. 1) compare well with those in similar related compounds. The piperidine ring is in the chair form with the N-methyl group and O(10) axially attached, thus requiring the epoxy group to be at the opposite side of the bridge. The configuration about C(13) is S. The carboxylic ester group has the usual conformation and the benzene ring is planar with mean C-C 1.386 Å. Packing is effected by normal van der Waals interactions.





(1S)-(1 $\alpha$ ,6 $\alpha$ R,7 $\alpha$ )-1,5,6,12-TETRAHYDRO-9-METHOXY-4-METHYL-14-OXO-1,7-ETHANO-4H,7H-FURO(4',3',2'.1,8)NAPHTH(1,2)AZOCINE-2-CARBOXYLIC ACID METHYL ESTER  
 $C_{22}H_{23}NO_5$

A. SINGH, S. ARCHER, K. HOOGSTEEN and J. HIRSHFIELD, 1982. J. Org. Chem., 47, 752-754.

Orthorhombic,  $P2_12_12_1$ ,  $a = 10.440$ ,  $b = 11.915$ ,  $c = 13.352$  Å,  $Z = 4$ . Cu radiation,  $R = 0.075$  for 1423 reflexions.

The structure of this derivative of thebaine was established by X-ray analysis, Fig. 1.

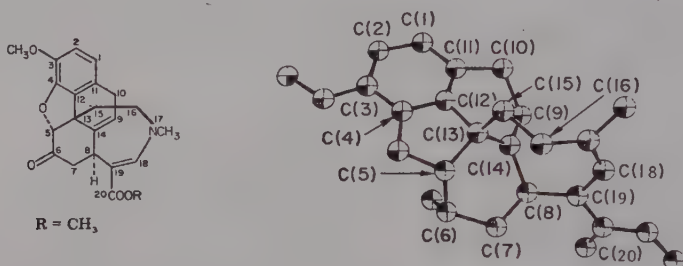


Fig. 1. Formula and view of the thebaine derivative,  $C_{22}H_{23}NO_5$ .

(-)-VINCADIFFORMIN DERIVATIVE HYDRATE

$C_{22}H_{28}N_2O_2 \cdot H_2O$

J. LAMOTTE, O. DIDEBERG, L. DUPONT and G. LEWIN, 1982. Acta Cryst., B38, 3133-3135.

Monoclinic,  $C2$ ,  $a = 19.112$ ,  $b = 11.301$ ,  $c = 9.625$  Å,  $\beta = 104.835^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.075$  for 1884 reflexions.

The analysis shows that as a result of reaction upon (-)-vincadifformin, the original C(20)-C(21) bond is broken. The reaction product is shown in Fig. 1.

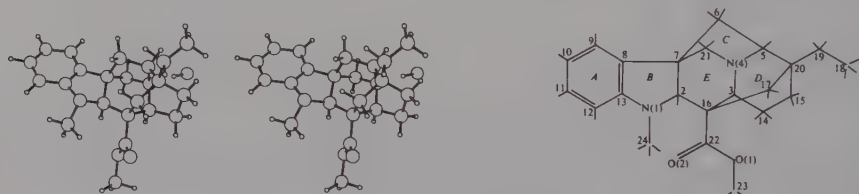


Fig. 1. A stereoview of  $C_{22}H_{28}N_2O_2$  and the numbering scheme.

COLCHICEINE ACETATE ETHYL ACETATE

$C_{23}H_{25}NO_7 \cdot 0.5C_4H_8O_2$

C. MIRAVITLLES, X. SOLANS, A. BLADÉ-FONT, G. GERMAIN and J. DECLERCQ, 1982. Acta Cryst., B38, 1782-1786.

Orthorhombic,  $P2_12_12_1$ ,  $a = 9.299$ ,  $b = 10.534$ ,  $c = 27.084$  Å,  $D_m = 1.16$ ,  $Z = 4$ . Mo radiation,  $R = 0.077$  for 1552 reflexions.

The molecule (Fig. 1) belongs to the isocolchicine series and has the acetamido O atom disordered. The troponoid ring C has a shallow, skew-boat conformation with the angle between the planes C(9),C(10),C(11) and C(9),C(11),C(12),C(8)  $3.8^\circ$  and



The alkaloid (Fig. 1) forms a ridged surface almost perpendicular to the b-axis with y coordinates ranging over only one eighth of the unit cell, and it is fully extended along the a-c diagonal. This extended geometry results from the trans fusion of the rings joined by C(5)-C(10) which allows C(24)H<sub>3</sub> and C(25)H<sub>3</sub> to occupy axial positions with a separation of 3.213 Å. The C(3)-O(21) bond distance (1.192 Å) confirms that this is a carbonyl group and its intra-annular angle C(2)-C(3)-C(4) is 123.2° and the adjacent angle at C(2) is also enlarged (120.6°). At C(5) all the C-C-C angles exceed the tetrahedral value, squeezing the 5-H atom. The saturated heterocyclic ring is distorted by angle deformation at the ring junction with the indole fragment, with N(13)-C(8)-C(9) 97.2 and C(12)-C(11)-C(9) 100.4°.

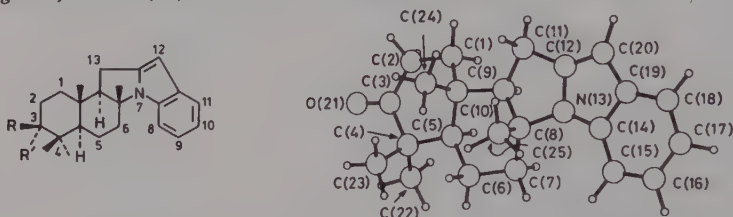


Fig. 1. Molecular skeleton (RR' = O) and structure of C<sub>23</sub>H<sub>29</sub>NO.

6α-HYDROXY-5α-CYANO-2α,7α-DIHYDROCORYNANTHEIDINE

C<sub>23</sub>H<sub>29</sub>N<sub>3</sub>O<sub>4</sub>

D. HERLEM, A. FLORES-PARRA, F. KHUONG-HUU, A. CHIARONI and C. RICHE, 1982. Tetrahedron, 38, 271-278.

Orthorhombic, P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, a = 26.719, b = 10.032, c = 8.119 Å, Z = 4. Cu radiation, R = 0.071 for 1484 reflexions.

The structure of this reaction product is shown in Fig. 1.

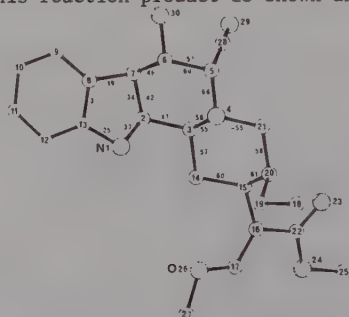


Fig. 1. A view of C<sub>23</sub>H<sub>29</sub>N<sub>3</sub>O<sub>4</sub> with torsion angles.

DELPHINIFOLINE

C<sub>23</sub>H<sub>37</sub>NO<sub>7</sub>

K.A. KERR and P.W. CODDING, 1982. Acta Cryst., B38, 1237-1241.

Orthorhombic, P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, a = 15.750, b = 16.356, c = 8.415 Å, Z = 4. Mo radiation, R = 0.038 for 1154 reflexions.

Delphinifoline is established as a member of the lycoctonine family of alkaloids by the presence of an oxygenated functional group at C(7) (Fig. 1), but the α configuration observed at C(1) is unexpected and calls into question the structural assignments of all alkaloids of this class.

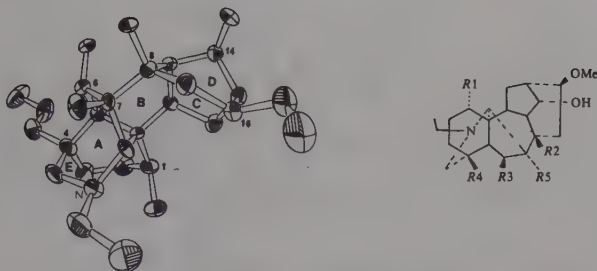


Fig. 1.  $C_{23}H_{37}NO_7$ : a perspective view of the molecule and the chemical formula ( $R_1 = OH$ ,  $R_2 = R_3 = R_5 = OH$ ,  $R_4 = CH_2OCH_3$ ).

#### O-DIACETYLGOUREGINE

$C_{24}H_{23}NO_7$

M. LEBOEUF, D. CORTES, R. HOCQUEMILLER, A. CHIARONI and C. RICHE, 1982. *Tetrahedron*, **38**, 2889-2896.

Orthorhombic,  $Pbcn$ ,  $a = 18.171$ ,  $b = 8.175$ ,  $c = 29.312 \text{ \AA}$ ,  $Z = 8$ .  $R = 0.055$ .

The structure of this new isoquinoline alkaloid is shown in Fig. 1. The dihedral angle between the two aromatic rings is  $126^\circ$ .

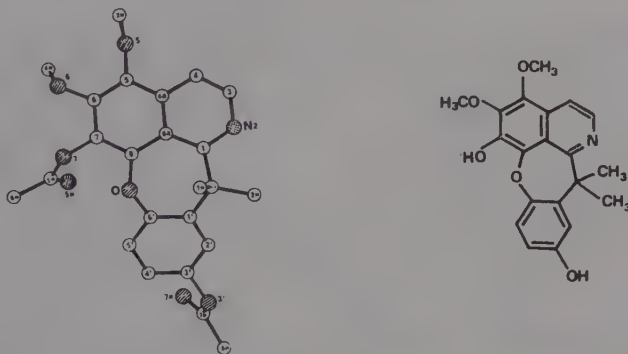


Fig. 1. A view of  $C_{24}H_{23}NO_7$  and gouregine.

#### LYCOCTONINE KETO LACTAM ACID

$C_{24}H_{33}NO_8$

M. CYGLER, M. PRZYBYLSKA and O.E. EDWARDS, 1982. *Acta Cryst.*, **B38**, 1500-1503.

Orthorhombic,  $P2_12_12_1$ ,  $a = 13.810$ ,  $b = 15.527$ ,  $c = 10.644 \text{ \AA}$ ,  $D_m = 1.341$ ,  $Z = 4$ . Cu radiation,  $R = 0.037$  for 2475 reflexions.

The molecule (Fig. 1) contains a strong, intramolecular hydrogen bond ( $O(2) \dots O(4)$   $2.503(3) \text{ \AA}$ ). The configuration at  $C(1)$  is the inverse of that previously accepted for related compounds. The methyl group at  $O(8)$  is disordered. All rings in the molecule are cis fused. The groups  $C(4), C(19), N, O(4)$  and  $O(2), C(3), C(4), O(3)$  are planar but  $C(6), C(7), C(8), O(6)$  is not.

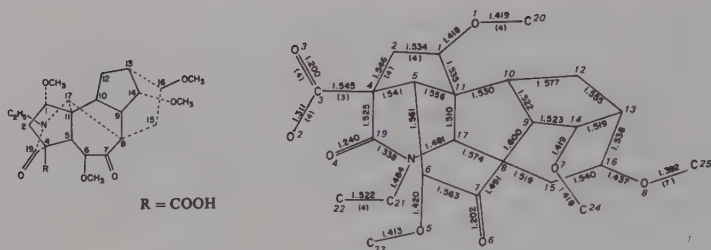


Fig. 1. Bond lengths and torsion angles in  $\text{C}_{24}\text{H}_{33}\text{NO}_8$ .

4-AMINO-4-DEOXYMETHYLENEANHYDROLYCOCTONAM ACETOXY LACTAM ACID  
 $\text{C}_{24}\text{H}_{35}\text{NO}_8$

M. CYGLER, M. PRZYBYLSKA and O.E. EDWARDS, 1982. *Acta Cryst.*, **B38**, 479-482.

Orthorhombic,  $\text{P2}_12_12_1$ ,  $a = 12.480$ ,  $b = 21.025$ ,  $c = 9.512$  Å,  $D_m = 1.233$ ,  $Z = 4$ . Cu radiation,  $R = 0.048$  for 2158 reflexions.

O(6)-H...O(4) hydrogen bonds of length 2.619(4) Å link the molecules (Fig. 1) into spirals along  $c$ . The A/B, B/C, C/D and C/E ring junctions are cis fused with rings A and B having half-chair, and C and D envelope conformations. The D and E rings form a bicyclo[3.2.1]octane system.

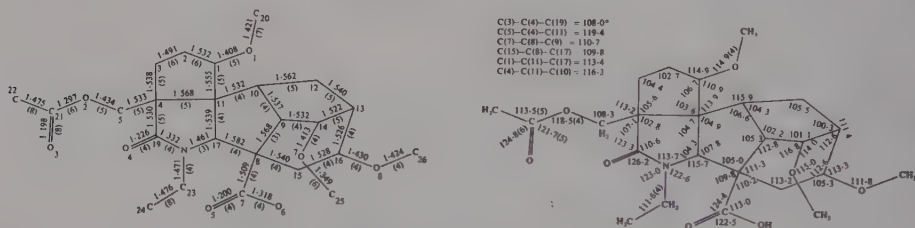


Fig. 1. Bond lengths and angles in  $\text{C}_{24}\text{H}_{35}\text{NO}_8$ .

DES(OXYMETHYLENE)LYCOTONINE HYDROIODIDE HYDRATE  
 $\text{C}_{24}\text{H}_{40}\text{INO}_6 \cdot \text{H}_2\text{O}$

O.E. EDWARDS and M. PRZYBYLSKA, 1982. *Canad. J. Chem.*, **60**, 2661-2667.

Monoclinic,  $\text{P2}_1$ ,  $a = 12.758$ ,  $b = 9.415$ ,  $c = 10.990$  Å,  $\beta = 97.81^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.085$  for 2012 reflexions.

The analysis corrects the structure originally deduced from two-dimensional projection studies (1), and shows that the 1-methoxyl group is  $\alpha$  (Fig. 1).

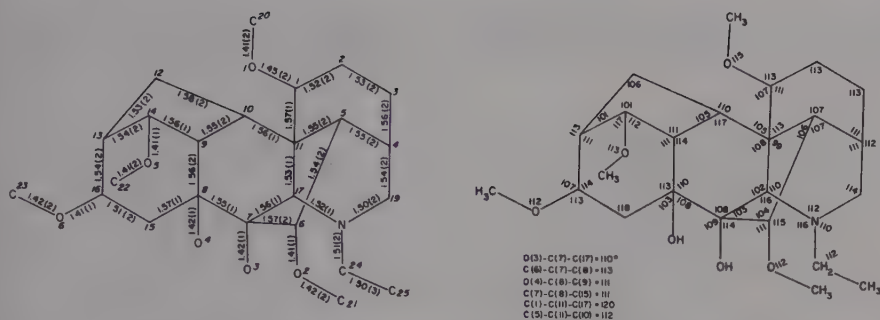
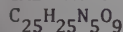


Fig. 1. Bond lengths and angles in des(oxymethylene)lycotonine hydroiodide.

# 1. Structure Reports, 26, 795.

## CALEBASSININE-1 PICRATE



A. GUGGISBERG, R. PREWO, J.H. BIERI and M. HESSE, 1982. *Helv. Chim. Acta*, **65**, 2587-2597.

Monoclinic,  $P2_1$ ,  $a = 7.080$ ,  $b = 13.049$ ,  $c = 12.889$  Å,  $\beta = 92.91^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.067$  for 5416 reflexions (at  $-140^\circ\text{C}$ ).

The analysis establishes that the calebassinine-1 cation has the structure and relative configuration shown in Fig. 1. The ions are linked by O-H...O (2.671 Å) and C-H...O (3.177 Å) hydrogen bonds.

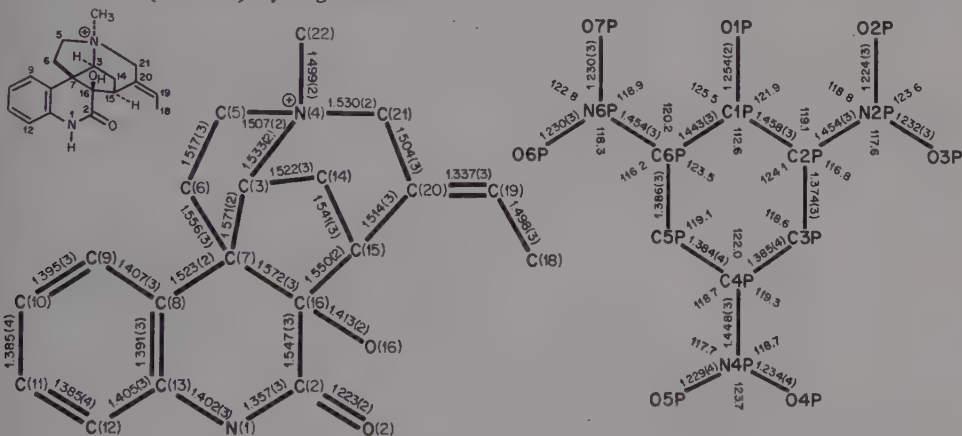


Fig. 1. The calebassinine-1 structure and dimensions in the cation and picrate anion.

## POLYAVOLENSIN



C.P. FALSHAW, T.J. KING and D.A. OKORIE, 1982. *Tetrahedron*, **38**, 2311-2313.

Hexagonal,  $P6_322$ ,  $a = 10.187$ ,  $c = 71.58$  Å,  $Z = 12$ . Mo radiation,  $R = 0.0665$  for 1107 reflexions.



Polyavolensin is determined to have the structure shown in Fig. 1. The pyrrolidine ring has an envelope conformation, while the trans-fused decalin system is chair-chair.

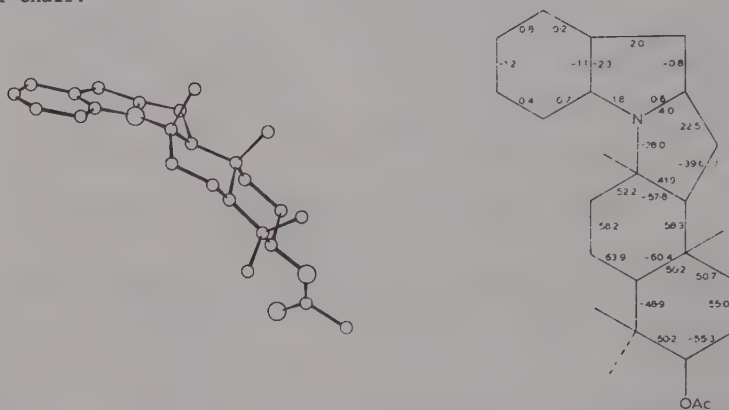
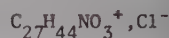
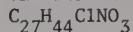


Fig. 1. Polyavolensin and torsion angles.

#### VERTICINONE HYDROCHLORIDE



V. KETTMANN, I. MAŠTEROVÁ and J. TOMKO, 1982. Acta Cryst., B38, 978-980.

Orthorhombic,  $P2_12_12_1$ ,  $a = 10.311$ ,  $b = 10.869$ ,  $c = 23.564$  Å,  $D_m = 1.10$ ,  $Z = 4$ . Cu radiation,  $R = 0.050$  for 1231 reflexions.

In the cation (Fig. 1) all the six-membered rings are in the chair conformation and the five-membered ring is in the envelope conformation. Ring fusions are A/B trans, B/C trans, C/D cis, D/E trans and E/F trans. The crystal structure consists of discrete ions held together by ionic and van der Waals forces. The chloride ion is situated so as to form three hydrogen bonds (one  $\text{N}^+-\text{H}\cdots\text{Cl}^-$  and two  $\text{O}-\text{H}\cdots\text{Cl}^-$ ) to three different ions of verticinone.

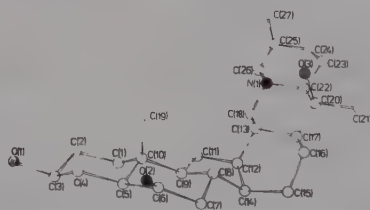
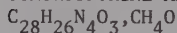


Fig. 1. Verticinone hydrochloride: a perspective view of the cation.

#### STAUROSPORINE METHANOL SOLVATE



A. FURUSAKI, N. HASHIBA, T. MATSUMOTO, A. HIRANO, Y. IWAI and S. OMURA, 1982. Bull. Chem. Soc. Jpn., 55, 3681-3685.

Monoclinic,  $C2$ ,  $a = 23.487$ ,  $b = 7.636$ ,  $c = 15.638$  Å,  $\beta = 116.71$ ,  $Z = 4$ . Cu radiation,  $R = 0.047$  for 2532 reflexions.

The analysis establishes the structure of this alkaloid (Fig. 1) and shows that it contains a dimeric indole structure. Because of steric hindrance, the indole[2,3-a]carbazole aromatic system is slightly bent. The two terminal benzene rings make an angle of  $7.5^\circ$  with each other.

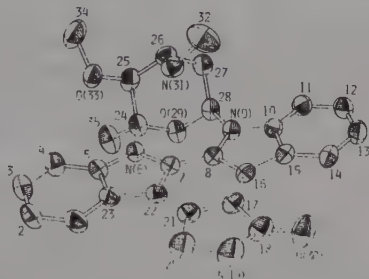
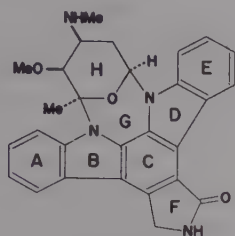


Fig. 1. The  $C_{28}H_{26}N_4O_3$  molecule.

#### LYTHRANINE DIMETHYL SULPHOXIDE MONOHYDRATE

$C_{28}H_{37}NO_5 \cdot C_2H_6OS \cdot H_2O$

P.J. COX and G.A. SIM, 1982. *Acta Cryst.*, B38, 303-306.

Monoclinic,  $P2_1$ ,  $a = 12.227$ ,  $b = 9.969$ ,  $c = 12.528$  Å,  $\beta = 92.00^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.044$  for 2895 reflexions.

In the crystal structure (Fig. 1) the lythranine, dimethyl sulphoxide and water molecules are linked together by hydrogen bonds:  $O \dots O$  2.67 and 2.82 Å,  $O \dots N$  2.74 Å. The lythranine molecule has a molecular conformation very similar to that of bromolythranine (1). The dihedral angle between the benzene planes is  $66^\circ$  and the  $O(1) \dots O(2)$  distance 3.09 Å. The piperidine ring has a chair conformation. The dimethyl sulphoxide molecule is nonplanar, with S displaced 0.72 Å from the plane through the O and C atoms.

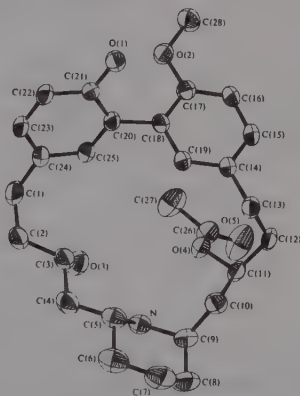
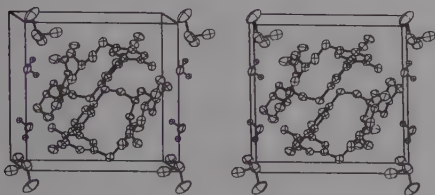


Fig. 1.  $C_{30}H_{45}NO_7S$ : a stereoview of the crystal structure, and a view of a lythranine molecule.

#### 1. Structure Reports, 38B, 586.

4-{8-ETHOXY-10,12-DIMETHYL-13-(2-QUINOLYL)-8-AZATRICYCLO[7.3.1.0<sup>2,7</sup>]TRIDECA-2(7),3,5-TRIEN-11-YLIDENE}MORPHOLINIUM IODIDE  
 $C_{29}H_{34}IN_3O_2$

T. UECHI, I. UEDA, H. NODA and M. HAMANA, 1982. *Acta Cryst.*, **B38**, 1848-1851.

Orthorhombic,  $P2_12_12_1$ ,  $a = 16.284$ ,  $b = 18.798$ ,  $c = 9.156$  Å,  $D_m = 1.38$ ,  $Z = 4$ . Mo radiation,  $R = 0.051$  for 2101 reflexions.

The structure contains discrete cations and anions. The almost planar quinoline ring makes an angle of about  $85^\circ$  with the benzene ring (Fig. 1). The azatricyclo-[7.3.1.0<sup>2,7</sup>]trideca-2(7),3,5-triene fragment has a twin-chair conformation with appreciably distorted rings while the morpholine ring has a chair conformation.

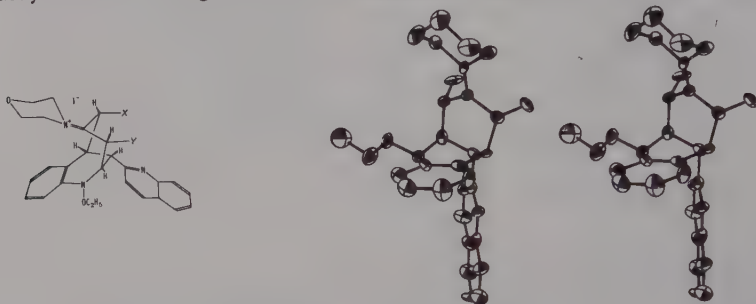
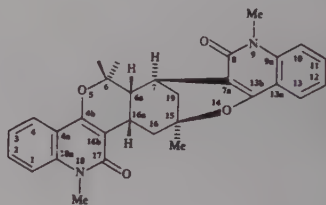


Fig. 1. The molecular skeleton ( $X = Y = \text{CH}_3$ ) and a stereoview of the  $\text{C}_{29}\text{H}_{34}\text{IN}_3\text{O}_2$  cation.

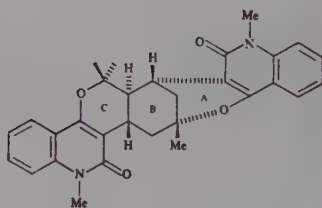
#### PARAENSIDIMERIN A

$\text{C}_{30}\text{H}_{30}\text{N}_2\text{O}_4$  (I)



#### PARAENSIDIMERIN C

$\text{C}_{30}\text{H}_{30}\text{N}_2\text{O}_4$  (II)



L. JURD, R.Y. WONG and M. BENSON, 1982. *Aust. J. Chem.*, **35**, 2505-2517.

I. Monoclinic,  $P2_1/n$ ,  $a = 9.491$ ,  $b = 22.131$ ,  $c = 11.554$  Å,  $\beta = 96.17^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.154$  for 2282 reflexions.

II. Tetragonal,  $I4_1/a$ ,  $a = 34.520$ ,  $c = 8.775$  Å,  $Z = 16$ . Cu radiation,  $R = 0.170$  for 2038 reflexions.

The structures of the two isomers were established to be those shown above. The low accuracy of the analysis precluded any detailed structural discussion.

## PYRODELPHININE

 $C_{31}H_{41}NO_7$  (I)

## DELPHININE

 $C_{33}H_{45}NO_9$  (II)

S.W. PELLETIER, J. FINER-MOORE, R.C. DESAI, N.V. MODY and H.K. DESAI, 1982. *J. Org. Chem.*, **47**, 5290-5297.

I. Monoclinic,  $P2_1$ ,  $a = 12.415$ ,  $b = 8.680$ ,  $c = 13.617$  Å,  $\beta = 104.143^\circ$ ,  $D_m = 1.29$ ,  $Z = 2$ . Cu radiation,  $R = 0.036$  for 1930 reflexions.

II. Orthorhombic,  $P2_12_12_1$ ,  $a = 12.361$ ,  $b = 15.784$ ,  $c = 16.073$  Å,  $D_m = 1.29$ ,  $Z = 4$ . Mo radiation,  $R = 0.043$  for 1408 reflexions.

The structures of I and II (Fig. 1) are compared to deduce that resonance forms do not contribute to the ground state of I. It is suggested for I that in the electronic ground state of the molecule the free electron pair of the nitrogen, the C(17)-C(7)  $\sigma$  bond, and the  $\pi$ -electron pair of the C(8)-C(15) double bond are not conjugated. Rings A, C, E, and F have the same conformations in both molecules. The differences between the two molecules in the conformations of rings B and D can be attributed to the effect of the C(8)-C(15) double bond in pyrodelphinine. The D ring of delphinine is in a bent-chair conformation with atoms C(8), C(9), C(13), C(15), and C(16) nearly coplanar and C(14) forming the flap. In pyrodelphinine ring D has a half-chair conformation, flattened at the double bond end.

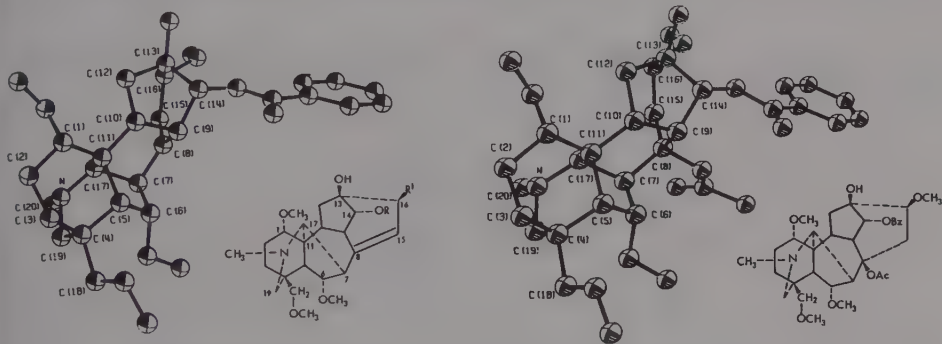


Fig. 1. Pyrodelphinine ( $R = Bz$ ,  $R' = OMe$ ) (left) and delphinine (right).

## ACONITINE

 $C_{34}H_{47}NO_{11}$ 

P.W. CODDING, 1982. *Acta Cryst.*, **B38**, 2519-2522.

Orthorhombic,  $P2_12_12_1$ ,  $a = 15.601$ ,  $b = 17.069$ ,  $c = 12.248$  Å,  $Z = 4$ . Mo radiation,  $R = 0.072$  for 2322 reflexions.

In the molecule (Fig. 1) rings A, B and E are in chair form, ring C is an envelope with C(14) at the flap, ring D is a boat with the end at C(15) flattened and ring F is a half-chair. There are two O-H...O intramolecular hydrogen bonds, with O...O 2.774(7) and 2.575(5) Å, in the molecule. No intermolecular hydrogen bonds occur and the packing appears to be determined by van der Waals contacts.

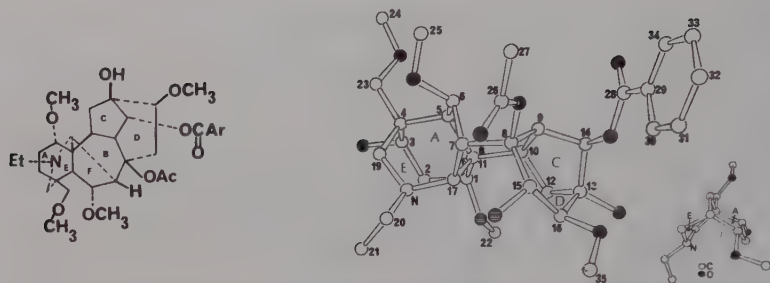
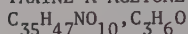


Fig. 1.  $C_{34}H_{47}NO_{11}$ : molecular structure and perspective drawing of the molecule, with insert (right) showing the chair-chair junction of rings A and E.

#### TAXINE A ACETONE SOLVATE



E. GRAF, A. KIRFEL, G.-J. WOLFF and E. BREITMAIER, 1982. Justus Liebigs Ann. Chem., 376-381.

Monoclinic,  $P2_1$ ,  $a = 10.860$ ,  $b = 12.808$ ,  $c = 13.666$  Å,  $Z = 2$ . Mo radiation,  $R = 0.061$  for 2256 reflexions.

Fig. 1 gives the bond lengths found for the alkaloid. There is an intramolecular hydrogen bond ( $O-H \cdots O$  2.497 Å) between the hydroxy and keto groups on the ten-membered ring.

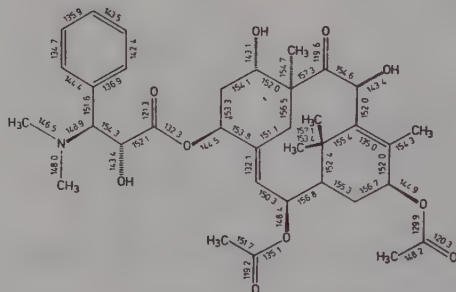
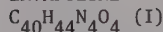
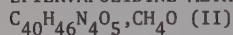


Fig. 1. Schematic view of taxine A with bond lengths (pm).

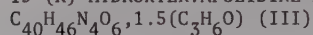
#### ERVAFOLINE



#### EPIERVAFOLIDINE METHANOL



#### 19'(R)-HYDROXYERVAFOLIDINE ACETONE



A. HENRIQUES, C. KAN, A. CHIARONI, C. RICHE, H.-P. HUSSON, S.-K. KAN and M. LOUNASMAA, 1982. J. Org. Chem., 47, 803-811.

I. Monoclinic,  $P2_1$ ,  $a = 10.326$ ,  $b = 14.738$ ,  $c = 11.645$  Å,  $\beta = 111.65^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.047$  for 2702 reflexions.

II. Orthorhombic,  $P2_12_12_1$ ,  $a = 24.469$ ,  $b = 18.246$ ,  $c = 8.064$  Å,  $Z = 4$ . Cu radiation,  $R = 0.077$  for 2677 reflexions.

III. Orthorhombic,  $P2_12_12_1$ ,  $a = 26.964$ ,  $b = 13.791$ ,  $c = 10.706$  Å,  $Z = 4$ . Cu radiation,  $R = 0.100$  for 2973 reflexions.

The analysis establishes the structure and configuration of these alkaloids to be as shown in Fig. 1.

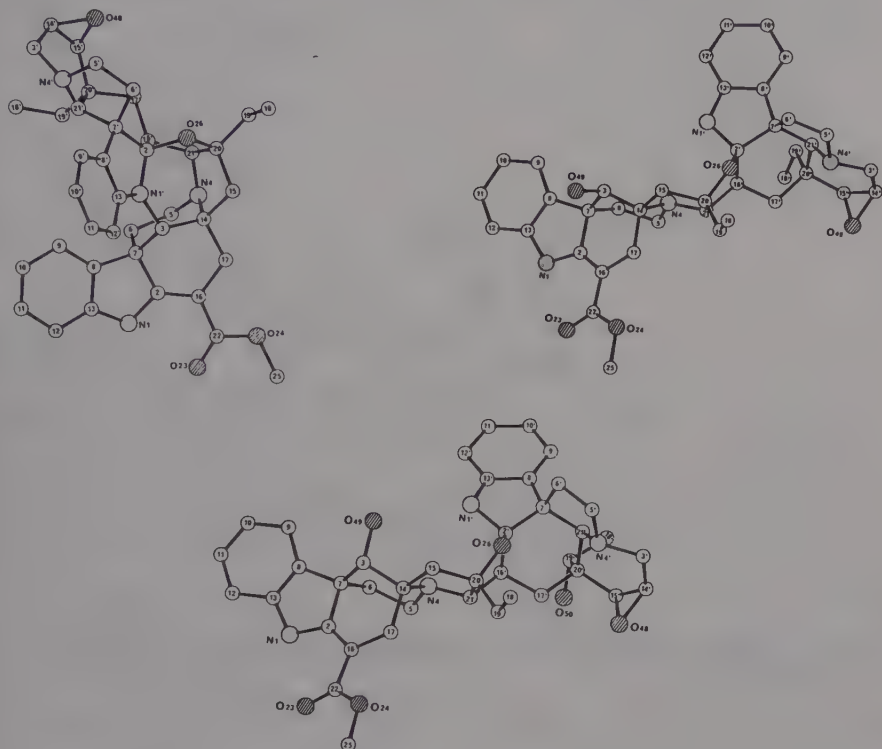


Fig. 1. Perspective views of ervafoline (top left), epiervafolidine (top right) and 19'-hydroxyervafolidine (centre).

#### GARDMULTINE METHANOL SOLVATE



J.V. SILVERTON and T. AKIYAMA, 1982. J. Chem. Soc. Perkin I, 1263-1267.

Orthorhombic,  $P2_12_12_1$ ,  $a = 8.625$ ,  $b = 20.791$ ,  $c = 24.494$  Å,  $Z = 4$ . Cu radiation,  $R = 0.053$  for 3238 reflexions.

Gardmultine (Fig. 1) is a novel bis-indole alkaloid isolated from "Gardneria multiflora", which consists of a gardneramine unit (g) and a chitosenine unit (c) linked by a spiro system. The bond lengths and angles agree well with expected values except for C(18g) through C(25g) which have e.s.d.'s of 0.01 Å. This is probably due to contamination of the crystals with the demethoxy compound. C(2c)-N(1c) is shorter (1.353) than the corresponding C(2g)-N(1g) bond (1.454 Å) due to extended resonance involving the former. There are no significantly short intermolecular distances in the crystal. Large columnar holes near the centre of the unit cell are partially filled with methanol molecules which are linked to O(1c) by a hydrogen bond (2.689 Å).





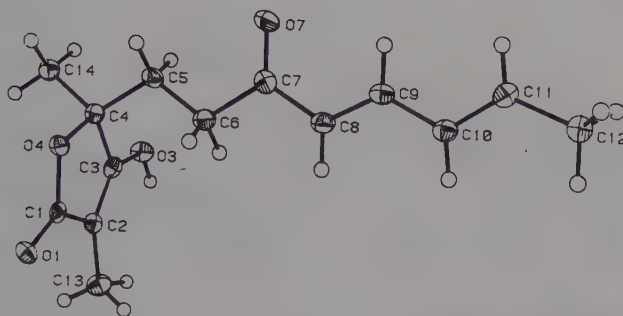
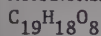


Fig. 1. Verlinolide.

## ATRANORINE



C. BRASSY, B. BACHET, B. BODO and D. MOLHO, 1982. *Acta Cryst.*, B38, 3126-3128.

Orthorhombic, *Pbca*,  $a = 12.099$ ,  $b = 15.408$ ,  $c = 18.801$  Å,  $Z = 8$ . Cu radiation,  $R = 0.066$  for 2696 reflexions.

The phenyl rings in the molecule (Fig. 1) are perpendicular to each other. There are two intramolecular O-H...O hydrogen bonds ( $\text{H}\cdots\text{O}$  1.64 and 1.67 Å).

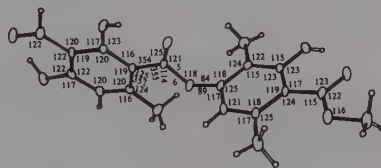
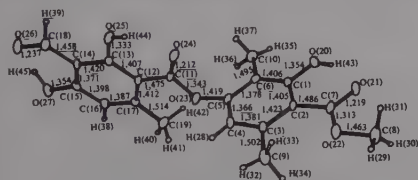
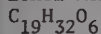


Fig. 1. Bond lengths and angles in atranorine.

## ETHYL MONATE C



J.P. CLAYTON, P.J. O'HANLON, N.H. ROGERS and T.J. KING, 1982. *J. Chem. Soc. Perkin I*, 2827-2833.

Monoclinic, *C2*,  $a = 26.278$ ,  $b = 7.105$ ,  $c = 11.205$  Å,  $\beta = 101.2^\circ$ ,  $D_m = 1.14$ ,  $Z = 4$ . Mo radiation,  $R = 0.066$  for 2190 reflexions. [Large crystal used for data collection.]

The X-ray analysis of the antibiotic derivative ethyl monate C (Fig. 1) confirmed the structure and the E-configuration of the 10-11 double bond. The C(10)-C(11) bond length is 1.308 and the C(2)-C(3) double bond conjugated to the ester function is 1.312 Å. The other bond lengths in the molecule are unexceptional. The bond angles within the ring are C(6)-C(7)-C(8) 112.1, C(5)-C(6)-C(7) 109.7, C(7)-C(8)-C(16) 109.0, C(8)-C(16)-O(5) 110.7, C(16)-O(5)-C(5) 112.4 and O(5)-C(5)-C(6) 109.2°.

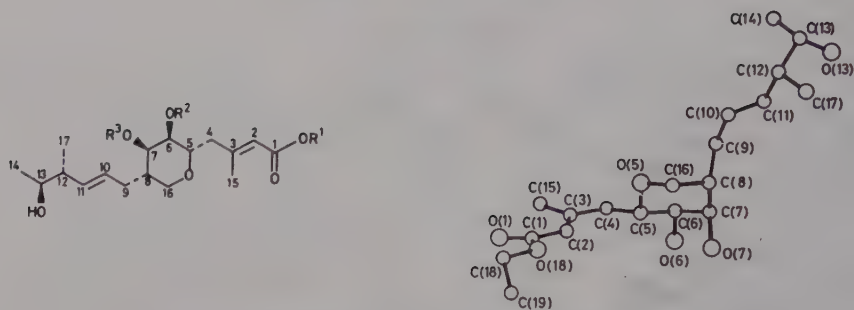


Fig. 1. The molecular skeleton ( $R^1 = \text{Et}$ ,  $R^2 = R^3 = \text{H}$ ) and structure of  $\text{C}_{19}\text{H}_{32}\text{O}_6$ .

1-(5-HYDROXY-2,2-DIMETHYL-2H-1-BENZOPYRAN-6-YL)-3-(4-HYDROXYPHENYL)-2-PROPEN-1-ONE  
 $\text{C}_{20}\text{H}_{18}\text{O}_4$

K. TOMITA, 1982. Cryst. Struct. Comm., 11, 221-226.

Monoclinic,  $\text{P2}_1/\text{c}$ ,  $a = 9.164$ ,  $b = 15.763$ ,  $c = 11.871 \text{ \AA}$ ,  $\beta = 104.396^\circ$ ,  $D_m = 1.25$ ,  $Z = 4$ . Cu radiation,  $R = 0.062$  for 2187 reflexions.

The molecule (Fig. 1) is almost planar with the dihedral angle of  $9.2^\circ$  between A ring and B ring and of  $7.3^\circ$  between B ring and C ring. Two hydrogen bonds occur in the crystal structure; one intramolecular, between O(2) and O(3) ( $\text{O}\cdots\text{O} \ 2.523 \text{ \AA}$ ), and one intermolecular (O(3) and O(4),  $\text{O}\cdots\text{O} \ 2.729 \text{ \AA}$ ).

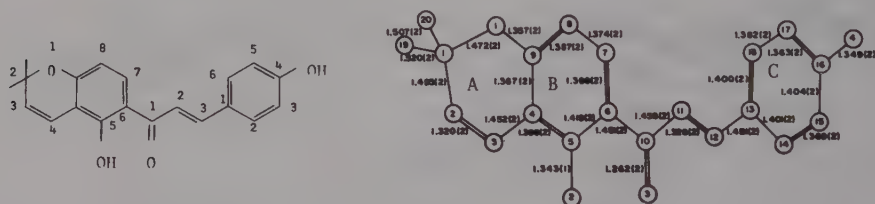


Fig. 1. The  $\text{C}_{20}\text{H}_{18}\text{O}_4$  structure.

TRACHYLOBAGIBBERELLIN  $\text{A}_{40}$  METHYL ESTER

$\text{C}_{20}\text{H}_{26}\text{O}_5$

B.M. FRAGA, A.G. GONZALEZ, M.G. HERNANDEZ, J.R. HANSON and P.B. HITCHCOCK, 1982. J. Chem. Soc. Chem. Commun., 594-595.

Orthorhombic,  $\text{P2}_12_12_1$ ,  $a = 8.298$ ,  $b = 10.612$ ,  $c = 20.152 \text{ \AA}$ ,  $Z = 4$ . Mo radiation,  $R = 0.063$  for 1006 reflexions.

The X-ray analysis established the molecular structure of the title compound (Fig. 1), a metabolite of a diterpenoid pentacyclic hydrocarbon.

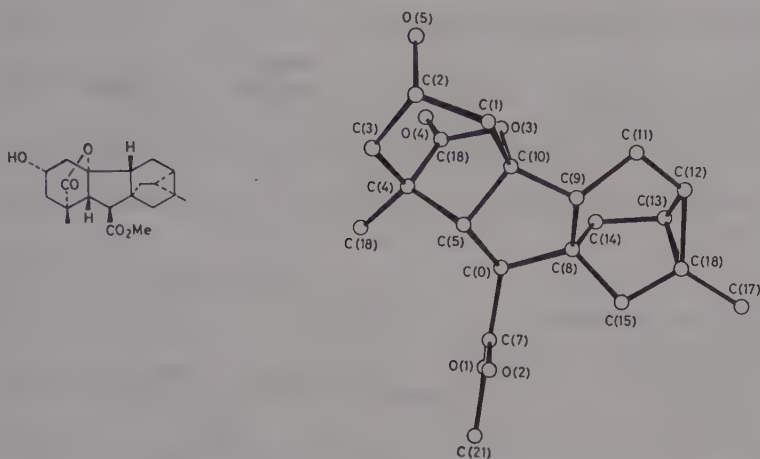


Fig. 1.  $C_{20}H_{26}O_5$ : the molecular skeleton and structure.

ENT-3 $\alpha$ ,10,14 $\beta$ ,16 $\beta$ -TETRAHYDROXY-13-METHYL-17,20-DINOR-8,13-EPI-GIBBERELLANE-7,19-DIOIC ACID 19,10-LACTON METHYLESTER

$C_{20}H_{28}O_7$

G. RECK, L. KUTSCHABSKY, G. ADAM and T. VAN SUNG, 1982. Cryst. Struct. Comm., **11**, 1989-1996.

Tetragonal,  $P4_32_1$ ,  $a = 13.765$ ,  $c = 22.552$  Å,  $Z = 8$ . Mo radiation,  $R = 0.099$  for 1944 reflexions.

A stereoview of the molecule is shown in Fig. 1. The six-membered rings A (C1-C2-C3-C4-C5-C10) and C (C8-C9-C11-C12-C13-C14) are distorted from the regular chair conformation. The chairs are flatter at C2 and C11, respectively, and sharper at C5 and C14, respectively. The asymmetry parameter  $\Delta C_5(C5)$  of  $0.7^\circ$  indicates an ideal envelope conformation for the lactone ring (C4-C5-C10-O10-C19) whereas the central five-membered ring B (C5-C6-C8-C9-C10) adopts (with  $\Delta C_2(C6)$   $1.2^\circ$ ) an almost ideal 9 $\beta$ ,10 $\alpha$ -half chair conformation. The third five-membered D ring (C8-C14-C13-C16-C15) is intermediate between 14 $\beta$ -envelope ( $\Delta C_5(C14)$   $7.8^\circ$ ) and 13 $\alpha$ ,14 $\beta$ -half chair ( $\Delta C_2(C15)$   $15.3^\circ$ ). The conformation of the 6 $\beta$ -methoxycarbonyl side chain is defined by the torsion angles C5-C6-C7-O7  $-54.0^\circ$  and C6-C7-O7O-C20  $175.4^\circ$ .

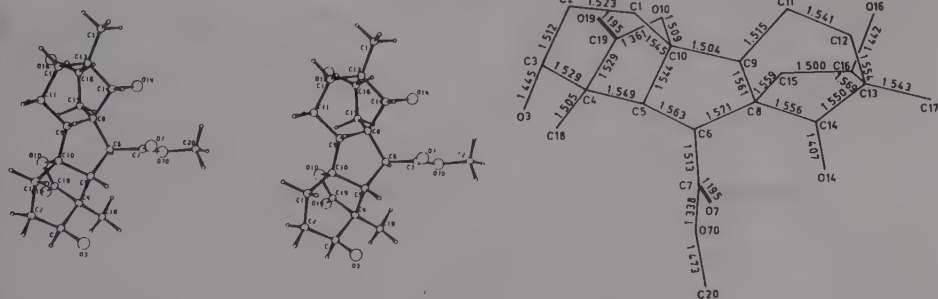


Fig. 1. A stereoview of  $C_{20}H_{28}O_7$  and bond lengths ( $\sigma$  0.016 Å).

10-NOR-9,11-SECOPROSTAGLANDIN F<sub>2</sub> METHYL ESTER  
C<sub>20</sub>H<sub>36</sub>O<sub>5</sub>

C.-H. LIN, D.L. ALEXANDER, C.G. CHIDESTER, R.R. GORMAN and R.A. JOHNSON, 1982. J. Am. Chem. Soc., 104, 1621-1628.

Orthorhombic, P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, a = 5.978, b = 8.815, c = 39.944 Å, Z = 4. R = 0.055.

The molecule has an overall "hairpin" appearance. Bond lengths and angles are shown in Fig. 1.

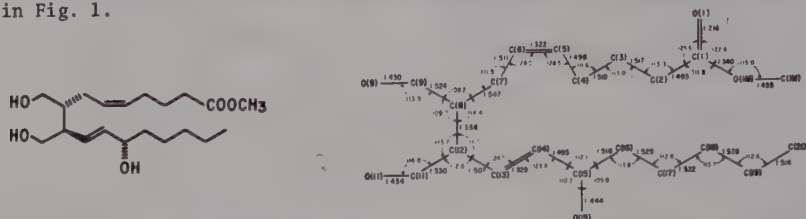


Fig. 1. Bond lengths and angles ( $\sigma = 0.004$ - $0.006$  Å,  $0.2$ - $0.3^\circ$ ) for C<sub>20</sub>H<sub>36</sub>O<sub>5</sub>.

DIMETHYL 3-DEHYDROGIBBERELLENATE  
C<sub>21</sub>H<sub>24</sub>O<sub>6</sub>

L. KUTSCHABSKY, D. PFEIFFER, G. ADAM and H.K. AL-EKABI, 1982. Cryst. Struct. Comm., 11, 2007-2012.

Monoclinic, P2<sub>1</sub>, a = 17.352, b = 7.713, c = 7.230 Å,  $\beta = 97.62^\circ$ , Z = 2. Mo radiation, R = 0.076 for 2371 reflexions.

In the molecule (Fig. 1) ring A adopts a 4 $\alpha$ ,5 $\beta$ -half-chair conformation and the five-membered ring B ( $\psi_m$  -16.6 and  $\Delta$  9.5°) approximates a 5 $\beta$ ,6 $\alpha$ -half chair conformation. The ring C is distorted from the regular chair being flatter at C(11) and sharper at C(14). The ring D ( $\psi_m$  50.0 and  $\Delta$  27.9°) approximates a 14 $\alpha$ -envelope conformation. The conformation of the 6 $\beta$ -methoxycarbonyl function is defined by the torsion angle C(5)-C(6)-C(7)-O(7) of 31.2°. The molecules are linked by O(13)-H...O(7') hydrogen bonds (2.811(6) Å).

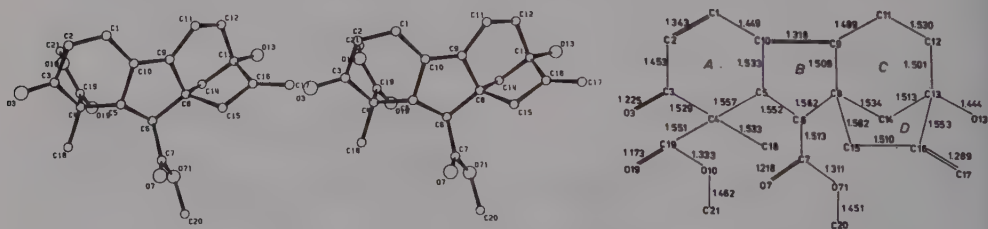
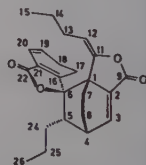


Fig. 1. A stereoview of C<sub>21</sub>H<sub>24</sub>O<sub>6</sub> and bond lengths ( $\sigma$  0.005-0.008 Å).

ANGEOLIDE  
C<sub>24</sub>H<sub>28</sub>O<sub>4</sub>



S.K. BANERJEE, B.D. GUPTA, W.S. SHELDRIK and G. HOFLE, 1982. Justus Liebigs Ann.

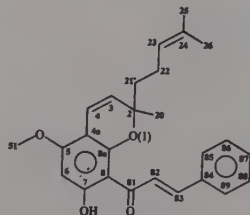
Chem., 699-707.

Monoclinic,  $P2_1/c$ ,  $a = 22.737$ ,  $b = 9.130$ ,  $c = 19.839$  Å,  $\beta = 91.43^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.068$  for 4564 reflexions.

The structure was confirmed to be that shown above with bond lengths and angles having expected values.

(±)-BOESENBERGIN A

$C_{26}H_{28}O_4$



T. JAIPETCH, S. KANGHAE, O. PANCHAROEN, J.A. PATRICK, V. REUTRAKUL, P. TUNTIWACH-WUTTIKUL and A.H. WHITE, 1982. Aust. J. Chem., 35, 351-361.

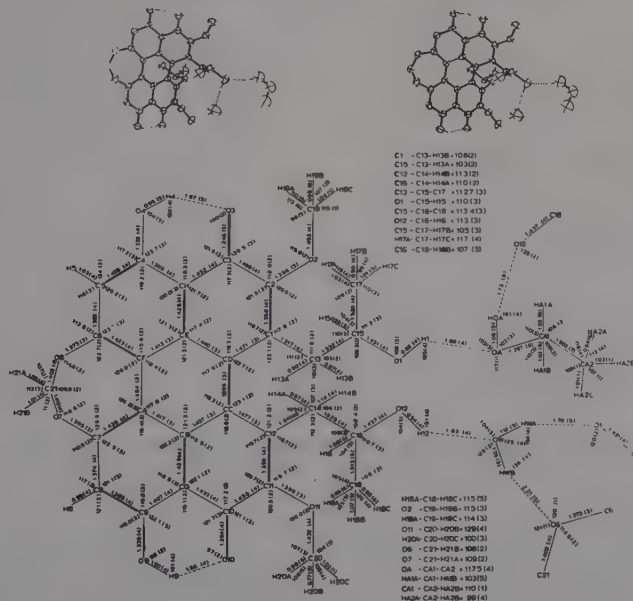
Monoclinic,  $P2_1/c$ ,  $a = 19.88$ ,  $b = 10.902$ ,  $c = 11.366$  Å,  $\beta = 91.90^\circ$ ,  $D_m = 1.21$ ,  $Z = 4$ . Mo radiation,  $R = 0.045$  for 1484 reflexions.

The structure of (±)-boesenbergin A was confirmed as that shown above with normal values for the bond lengths and angles.

1,12-BIS(2-HYDROXYPROPYL)-2,11-DIMETHOXY-6,7-METHYLENEDIOXY-4,9-DIHYDROXYPERYLENE-3,10-QUINONE ETHANOL WATER

$C_{29}H_{26}O_{10} \cdot C_2H_6O \cdot H_2O$

D. MENTZAFOS, A. TERZIS and S.E. FILIPPAKIS, 1982. Cryst. Struct. Comm., 11, 71-74.





Orthorhombic,  $P2_12_12_1$ ,  $a = 14.046$ ,  $b = 15.907$ ,  $c = 12.516 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.043$  for 2495 reflexions.

The analysis establishes the stereochemistry of this red phytotoxic pigment (Fig. 1). The molecule is not planar and there is a pseudo twofold rotation axis which does not pass through C(21). The water and ethanol molecules are linked to the pigment by O-H...O hydrogen bonds.

#### TERRITREM B

$C_{29}H_{34}O_9$

T.H. HSEU, C.K. YANG, K.H. LING, C.J. WANG and C.P. TANG, 1982. Cryst. Struct. Comm., 11, 199-206.

Orthorhombic,  $P2_12_12_1$ ,  $a = 8.681$ ,  $b = 12.477$ ,  $c = 25.314 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.091$  for 1674 reflexions.

The molecule (Fig. 1) appears as a long strip extended along the  $c$ -axis. The benzenoid ring A and pyrone ring B are essentially planar. The twist between the two planes is  $18(1)^\circ$ . Both the pyran ring C and cyclocitral ring E have a half-chair conformation. Their junctions with ring D are both trans. This results in ring D adopting the chair conformation. There are no intermolecular hydrogen bonds. The hydroxyl O9 atom is within hydrogen-bonding distance with O1 and O2 (O1...O9 2.70(2) and O2...O9 2.62(2)  $\text{\AA}$ ). An intramolecular hydrogen bond is found between O1 and O9. The bond angle at either oxygen atom in the lactone group and that of the oxygen atom in ring C are both substantially greater than normally expected. The placement of hydroxyl and carbonyl groups on the molecule is such that the molecule has on one side a nonpolar surface.

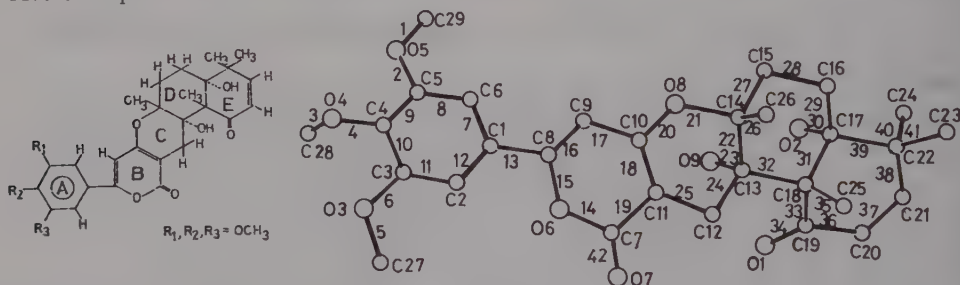


Fig. 1. A view of territrems B.

#### 7,11-EPOXY-THAPSIGARGIN

$C_{34}H_{48}O_{11}$

S.B. CHRISTENSEN, I.K. LARSEN, U. RASMUSSEN and C. CHRISTOPHERSEN, 1982. J. Org. Chem., 47, 649-652.

Monoclinic,  $P2_1$ ,  $a = 16.199$ ,  $b = 8.040$ ,  $c = 12.946 \text{ \AA}$ ,  $\beta = 100.84^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.080$  for 3889 reflexions (at 96 K).

The structures of thapsigargin and related sesquiterpene lactones have been determined from the X-ray analysis of the epoxy derivative, I. The conformation and relative configuration are shown in Fig. 1; the absolute configuration could not be deduced from the X-ray data. The ester groups at C(2) and C(10), the H-atom at C(1) and the 7,11-epoxide are all  $\alpha$ . The lactone ring is fused trans, and the ester groups at C(3) and C(8) and the C(13) methyl group are  $\beta$ . The cycloheptane ring adopts a distorted chair conformation, with C(7), C(8), C(1) and C(10) as the central plane. The cyclopentene ring and the lactone ring are slightly puckered, as shallow envelopes with C(3) and C(6) as the flaps.

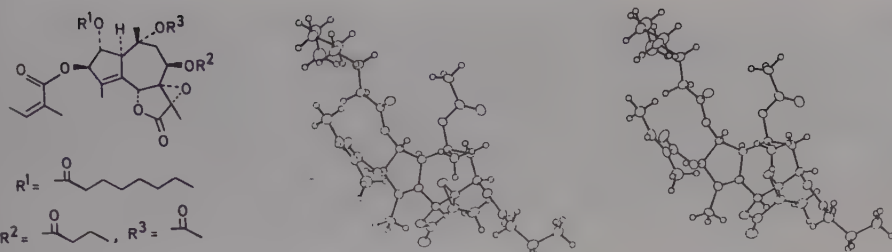


Fig. 1. Formula and stereoview of  $C_{34}H_{48}O_{11}$ .

(4Z,10Z,15Z)-2,3,7,8,12,13,17,18-OCTAETHYL-5-NITRO-21H,24H-BILIN-1,19-DIONE  
 $C_{35}H_{45}N_5O_4$  (I)

(4SR,5RS,10Z,15Z)-4,5-DIETHOXY-2,3,7,8,12,13,17,18-OCTAETHYL-4,5-DIHYDRO-21H,24H-BILIN-1,19-DIONE  
 $C_{39}H_{56}N_4O_4$  (II)

J.V. BONFIGLIO, R. BONNETT, D.G. BUCKLEY, D. HAMZETASH, M.B. HURTSHOUSE, K.M. ABDUL MALIK, S.C. NAITHANI and J. TROTTER, 1982. J. Chem. Soc. Perkin I, 1291-1302.

I. Triclinic,  $P\bar{1}$ ,  $a = 14.507$ ,  $b = 12.405$ ,  $c = 11.855$  Å,  $\alpha = 116.59$ ,  $\beta = 67.96$ ,  $\gamma = 114.22^\circ$ ,  $D_m = 1.16$ ,  $Z = 2$ . Cu radiation,  $R = 0.056$  for 3357 reflexions.

II. Monoclinic,  $P2_1/n$ ,  $a = 12.081$ ,  $b = 14.789$ ,  $c = 21.943$  Å,  $\beta = 100.79^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.081$  for 3636 reflexions.

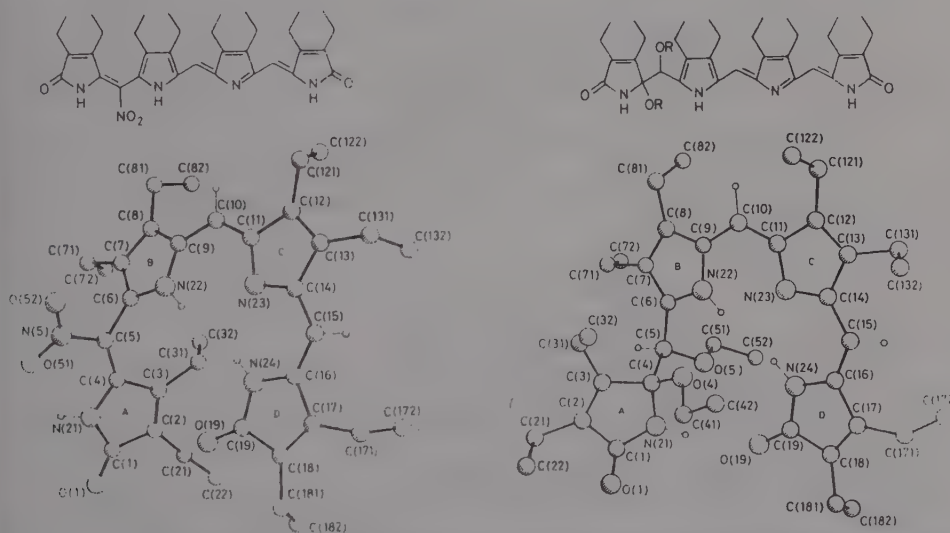


Fig. 1. Molecular skeletons and structures of  $C_{35}H_{45}N_5O_4$  (left) and  $C_{39}H_{56}N_4O_4$  ( $R = Et$ ) (right).

In (I) (Fig. 1) the pyrrole rings B, C and D are approximately co-planar but show a significant helical deviation from planarity. Ring A is planar and tilted at  $117^\circ$  from the mean plane of B, C, and D. The dihedral angle between the plane of the nitro group and the plane of C(5) and ring A is  $15.7^\circ$ . The conformation of the molecule is stabilised by an intramolecular hydrogen bond between O(51) and N(21)-H

(O(51)...N(21) 2.62 Å). There is marked bond localisation in the meso bridges; C(4)-C(5) 1.354, C(10)-C(11) 1.357, and C(15)-C(16) 1.351 Å, having considerable double bond character compared with C(5)-C(6) 1.465, C(9)-C(10) 1.422 and C(14)-C(15) 1.435 Å. The X-ray structure established the relative configuration of the ethoxy groups in II. Again the rings B, C and D are approximately co-planar with some helical character and ring A is rotated by 44.5° with respect to the planar portion. There is a short intramolecular contact of 3.20 Å between O(19) and N(21). There is little evidence of intermolecular hydrogen bonding in the crystal structure of I or II although there are a number of intermolecular contacts less than 3.5 Å.

#### 23-ACETOXY-28-BENZOATO-CERCOSPORIN

$C_{38}H_{32}O_{12}$

G. NASINI, L. MERLINI, G.D. ANDREETTI, G. BOCELLI and P. SGAROBOTTO, 1982. Tetrahedron, **38**, 2787-2796.

Orthorhombic,  $P2_12_12_1$ ,  $a = 12.327$ ,  $b = 15.552$ ,  $c = 17.074$  Å,  $Z = 4$ . Cu radiation,  $R = 0.0442$  for 2531 reflexions.

The six condensed rings are arranged in a helical fashion (Fig. 1) which is right-handed from the benzoate chain. In the perylene moiety, the delocalisation is limited, an alternation of short and long bonds is observed. The seven-membered has a boat conformation.

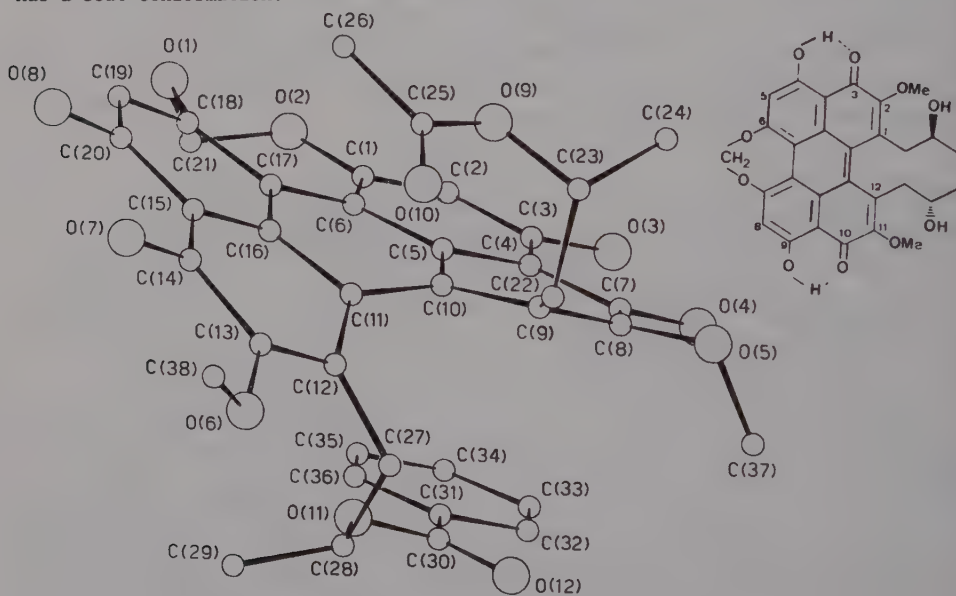


Fig. 1. The  $C_{38}H_{32}O_{12}$  molecule and cercosporin

#### BRYOSTATIN 1 METHANOL SOLVATE

$C_{47}H_{66}O_{17} \cdot x(CH_4O)$

G.R. PETTIT, C.L. HERALD, D.L. DOUBEK, D.L. HERALD, E. ARNOLD and J. CLARDY, 1982. J. Am. Chem. Soc., **104**, 6846-6848.

Orthorhombic,  $P2_12_12_1$ ,  $a = 21.782$ ,  $b = 20.428$ ,  $c = 23.664$  Å,  $Z = 8$ . Mo radiation,  $R = 0.07$  for 3553 reflexions (at -100°C).

The two independent molecules are virtually identical. The absolute configuration is 3(R),5(R),7(S),9(S),11(S),15(R),19(S),20(S),23(S),25(R),26(R) (Fig. 1). The molecule may be considered a macrolide 26-membered ring in which is embedded a 20-membered ring. All three pyran rings are in approximate chair conformations and each has a 4-substituent which points outward. All of the macrocycle substituents are equatorial with reference to the pyran rings.

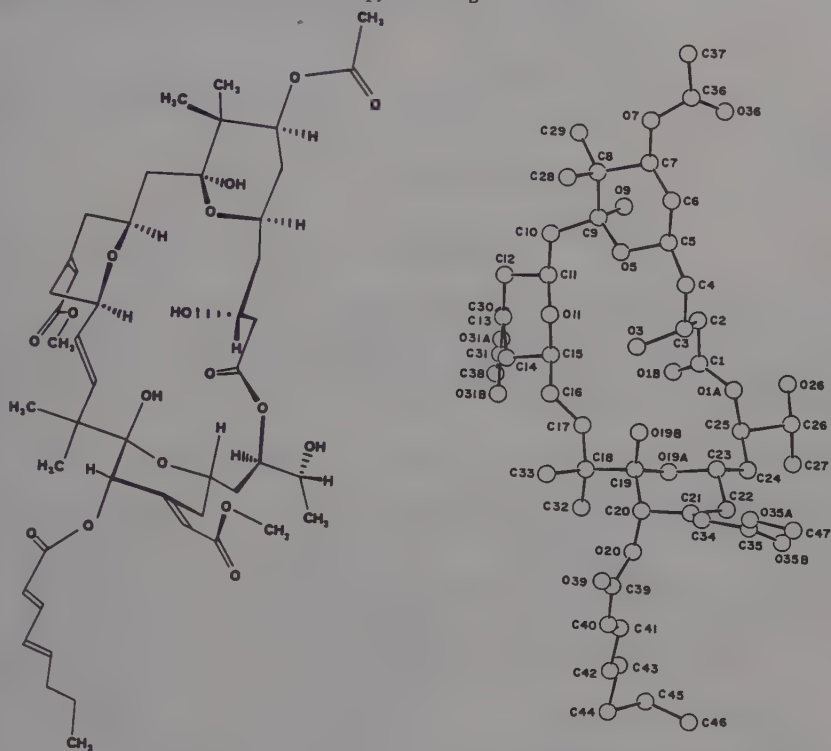
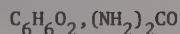


Fig. 1. Schematic (left) and perspective (right) drawings of bryostatin 1.

#### RESORCINOL UREA



M. PICKERING and R.W.H. SMALL, 1982. *Acta Cryst.*, **B38**, 3161-3163.

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.142$ ,  $b = 7.798$ ,  $c = 15.428$  Å,  $D_m = 1.321$ ,  $Z = 4$ . Mo radiation,  $R = 0.050$ .

The structure (Fig. 1) is a hydrogen-bonded complex, with O-H...O(urea) 2.696 and 2.679 Å and N-H...O(resorcinol) 2.944 and 3.128 Å. There are no short contacts between molecules of the same type.

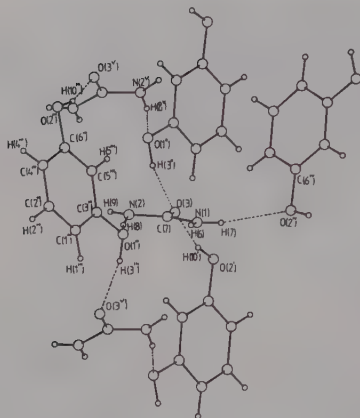


Fig. 1. A view of the resorcinol urea complex.

# 9-METHYLADENINE 2-THIOHYDANTOIN

$C_9H_{11}N_7OS$

$C_6H_7N_5 \cdot C_3H_4N_2OS$

R.E. CASSADY and S.W. HAWKINSON, 1982. Acta Cryst., B38, 2206-2209.

Monoclinic, C2/c,  $a = 21.140$ ,  $b = 7.085$ ,  $c = 16.101$  Å,  $\beta = 103.27^\circ$ ,  $D_m = 1.508$ ,  $Z = 8$ . Mo radiation,  $R = 0.035$  for 2475 reflexions.

The crystal structure (Fig. 1) consists of ribbons with alternating adenine and hydantoin moieties linked via (hydantoin)N-H...N(adenine) hydrogen bonds. The ribbons are held together via N-H...N hydrogen bonds involving the 9-methyladenine molecules. The sulphur and oxygen atoms of the 2-thiohydantoin molecules do not appear to act as acceptors in strong hydrogen bonds.

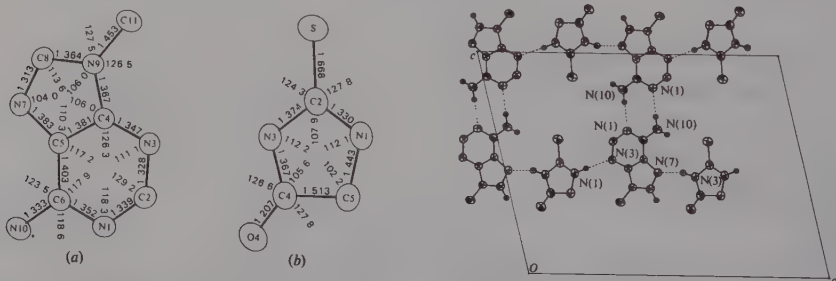


Fig. 1.  $C_6H_7N_5 \cdot C_3H_4N_2OS$ : bond lengths and angles for (a) 9-methyladenine and (b) 2-thiohydantoin, and hydrogen bonding in the complex.

# HEXAFLUOROBENZENE DEUTEROBENZENE

$C_{12}D_6F_6$

$C_6F_6 \cdot C_6D_6$

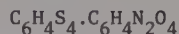
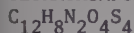
J.S.W. OVERELL and G.S. PAWLEY, 1982. Acta Cryst., B38, 1966-1972.

Rhombohedral,  $R\bar{3}m$  or  $R3m$ ,  $a = 7.310$ ,  $\alpha = 109.67^\circ$ ,  $Z = 1$ . Cu radiation,  $R = 0.098$  for 150 reflexions (at 279 K).

Molecules of each species are alternately arranged in infinite stacks along [111] with molecular planes perpendicular to this direction. Both constrained and

unconstrained refinements were carried out but failed to distinguish between models in which (a) only the carbon-deuterium pairs lie on mirror planes or (b) no atoms lie on mirror planes or whether in the former the R3m space group is favoured. On this last possibility the best estimate of the shift parameter leads to molecular pairing with a separation of 3.52 Å and with dimers separated by 3.71 Å. The main difference between models (a) and (b) is that in the former, molecules are eclipsed as viewed down the unique axis whereas in (b) they are staggered.

#### TETRATHIAFULVALENE p-DINITROBENZENE 1:1 COMPLEX



M.R. BRYCE, A.S. SECCO, J. TROTTER and L. WEILER, 1982. *Canad. J. Chem.*, **60**, 2057-2061.

Triclinic,  $P\bar{1}$ ,  $a = 6.915$ ,  $b = 7.615$ ,  $c = 8.149$  Å,  $\alpha = 79.39^\circ$ ,  $\beta = 69.55^\circ$ ,  $\gamma = 70.81^\circ$ ,  $Z = 1$ . Mo radiation,  $R = 0.028$  for 1222 reflexions.

Both component molecules lie on independent inversion centres and the crystal structure consists of molecules stacked in alternating donor-acceptor fashion along the c-axis (Fig. 1). Mixed sheets parallel to (012) are comprised of tetrathiafulvalene and p-dinitrobenzene molecules arranged end-on in the [021] direction. The intrastack interplanar spacing between TTF and DNB molecules averages 3.64 Å.

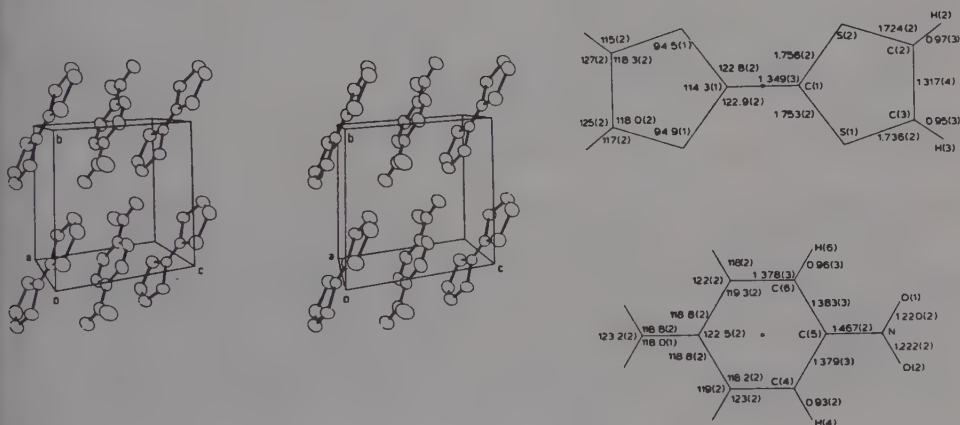


Fig. 1. A stereoview of the TTF:DNB complex and molecular dimensions.

#### 2,2'-BIS-1,3-DITHIOLE DIMETHYL DICYANOFUMARATE COMPLEX



J.E. MULVANEY, L. PANG, R.J. CRAMER and H.K. HALL, 1982. *J. Cryst. Spectrosc. Res.*, **11**, 331-342.

Monoclinic,  $P2_1/c$ ,  $a = 11.075$ ,  $b = 11.615$ ,  $c = 6.623$  Å,  $\beta = 95.7^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.0492$  for 404 reflexions.

In this complex (Fig. 1), the molecules lie on inversion centres and are contained in segregated parallel columns with a distance of 3.31 Å between dithiole molecules. Molecular dimensions are as expected.



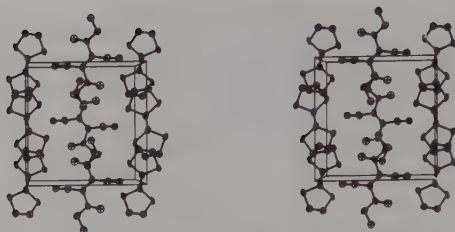


Fig. 1. A stereoview of the  $C_{14}H_{10}N_2O_4S_4$  complex.

DIBENZOTETRATHIAFULVALENE

$C_{14}H_{18}S_4$  (I)

DIBENZOTETRATHIOFULVALENE 7,7,8,8-TETRACYANO-p-QUINODIMETHANE

$C_{26}H_{12}N_4S_4$  (II)

$C_{14}H_8S_4, C_{12}H_4N_4$

DIBENZOTETRATHIOFULVALENE 2,5-DIFLUORO-7,7,8,8-TETRACYANO-p-QUINODIMETHANE

$C_{26}H_{20}F_2N_4S_4$  (III)

$C_{14}H_{18}S_4, C_{12}H_2F_2N_4$

T.J. EMGE, F.M. WIYGUL, J.S. CHAPPELL, A.N. BLOCH, J.P. FERRARIS, D.O. COWAN and T.J. KISTENMACHER, 1982. *Mol. Cryst. Liq. Cryst.*, **87**, 137-161.

I. Monoclinic,  $P2_1/c$ ,  $a = 12.082$ ,  $b = 3.955$ ,  $c = 14.553$  Å,  $\beta = 114.36^\circ$ ,  $D_m = 1.60$ ,  $Z = 2$ . Mo radiation,  $R = 0.036$  for 1852 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 9.215$ ,  $b = 10.644$ ,  $c = 7.734$  Å,  $\alpha = 113.32$ ,  $\beta = 122.28$ ,  $\gamma = 67.66^\circ$ ,  $D_m = 1.45$ ,  $Z = 1$ . Mo radiation,  $R = 0.070$  for 2402 reflexions.

III. Triclinic,  $P\bar{1}$ ,  $a = 8.838$ ,  $b = 9.276$ ,  $c = 7.794$  Å,  $\alpha = 101.29$ ,  $\beta = 100.89$ ,  $\gamma = 107.42^\circ$ ,  $D_m = 1.59$ ,  $Z = 1$ . Mo radiation,  $R = 0.040$  for 3687 reflexions.

In I, the molecules lie on inversion centres and the crystal structure contains columns of molecules, with the average molecular planes separated by 3.60 Å. In II and III, the components all lie on inversion centres and the crystal structures contain columns in which donor and acceptor molecules alternate with a mean separation of 3.4 Å. These DADA... stacked columns are tied into sheets through weaker donor-donor overlap. II and III differ in the way sheets of donor-coupled stacks are arranged relative to each other.

7,7,8,8-TETRACYANO-p-QUINODIMETHANE BIS(ETHANEDIAL DIOXIMATO)NICKEL(II) (1:1 ADDUCT)

$C_{16}H_{10}N_8NiO_4$

$C_4H_6N_4NiO_4, C_{12}H_4N_4$

M. MÉGNAMISI-BÉLOMBÉ and H. ENDRES, 1982. *Acta Cryst.*, **B38**, 1826-1828.

Monoclinic,  $P2_1/a$ ,  $a = 7.221$ ,  $b = 15.496$ ,  $c = 8.160$  Å,  $\beta = 102.54^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.041$  for 862 reflexions.

The structure consists of interpenetrating stacks of neutral, centrosymmetric Ni(II) complexes and TCNQ molecules along the diagonal of the xz plane in the  $[\bar{1}01]$  direction. There is a short contact (3.357(5) Å) between Ni and one of the two terminal nitrile N atoms at each end of the TCNQ molecule. The Ni complex (Fig. 1) is essentially planar while planes through the quinoid part and through the terminal  $C(CN)_2$  group of the TCNQ molecule form an angle of  $3.4^\circ$ .

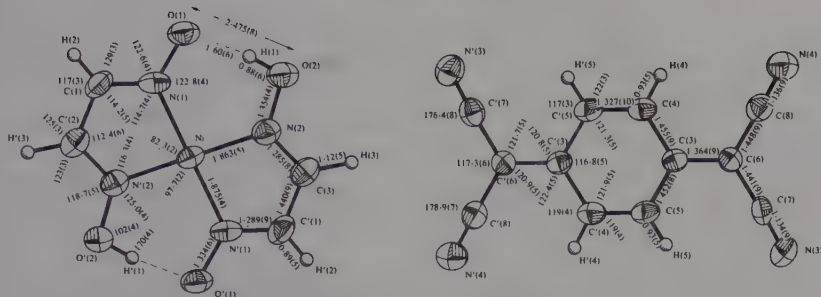


Fig. 1. The Ni complex and TCNQ molecule in  $C_{16}H_{10}N_8NiO_4$ .

BIS(2,4,6-TRIMETHYL-1-PYRIDYL) IODONIUM PERCHLORATE  
 $C_{16}H_{22}ClIN_2O_4$

$(C_8H_{11}N)_2I^+, ClO_4^-$

G.D. BRAYER and M.N.G. JAMES, 1982. *Acta Cryst.*, **B38**, 654-657.

Monoclinic,  $C2/c$ ,  $a = 27.844$ ,  $b = 11.036$ ,  $c = 23.412$  Å,  $\beta = 126.47^\circ$ ,  $D_m = 1.60$ ,  $Z = 12$ . Cu radiation,  $R = 0.057$  for 2171 reflexions.

The structure, with 1.5 formula units per asymmetric unit, contains one cation in a general position exhibiting a twist conformation of the two ring planes about I (dihedral angle  $35.7^\circ$ ) whereas the cation on a centre of symmetry has the two ring planes parallel but separated by 1.34 Å. I(1) is 0.142 and 0.262 Å from planes 1 and 2 respectively while I(2) is 0.674 Å from plane 3 (Fig. 1). One perchlorate ion lies on a twofold axis and is disordered.

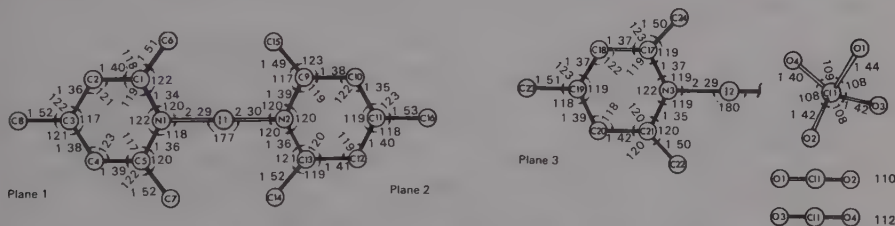


Fig. 1. Bond distances and angles in bis(2,4,6-trimethyl-1-pyridyl)iodonium perchlorate.

TETRATHIAFULVALENE TETRAFLUOROTETRACYANO-p-QUINODIMETHANE CYCLOHEXANE SOLVATE  
 $C_{18}H_4F_4N_4S_4 \cdot C_6H_{12}$

TTF·TCNQF<sub>4</sub>·C<sub>6</sub>H<sub>12</sub>

F.M. WIYGUL, T.J. EMGE and T.J. KISTENMACHER, 1982. *Mol. Cryst. Liq. Cryst.*, **90**, 163-171.

Monoclinic,  $P2/m$ ,  $a = 16.562$ ,  $b = 10.728$ ,  $c = 7.013$  Å,  $\beta = 97.39^\circ$ ,  $D_m = 1.57$ ,  $Z = 2$ . Mo radiation,  $R = 0.18$  for 2107 reflexions.

The components of this complex lie across crystallographic mirror planes and the crystal structure (Fig. 1) consists of segregated stacks of donors and acceptors propagating along the  $c$  axis. Parallel to these columnar arrays are solvent channels of unresolved composition. The TCNQF<sub>4</sub> acceptor column is both distance and overlap modulated (eclipsed, dimeric interaction at a mean separation of 3.25 Å, and ring-over-bond, monomeric interaction at 3.43 Å). The TTF donor column shows analogous eclipsed and ring-over-bond overlap patterns; however, the distance alternation (3.49 Å and 3.51 Å respectively) is within experimental error ( $\pm 0.02$  Å in each case).

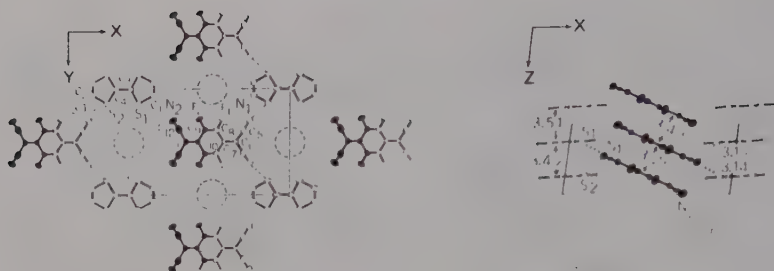
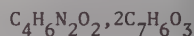
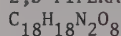


Fig. 1. The (001) and (010) projections for the TTF.TCNQF4.C<sub>6</sub>H<sub>12</sub> crystal structure.

2,5-PIPERAZINEDIONE - SALICYCLIC ACID (1:2 COMPLEX)



K.I. VARUGHESE and G. KARTHA, 1982. Acta Cryst., B38, 301-302.

Monoclinic, P2<sub>1</sub>/a, a = 18.52, b = 5.455, c = 8.787 Å, β = 104.28°, Z = 2. Cu radiation, R = 0.041 for 1397 reflexions.

In the complex (Fig. 1) the molecules are linked by hydrogen bonds with N...O(1) 2.844 Å and O(2)...O 2.592 Å. There is also an intramolecular hydrogen bond with O(3)...O(1) 2.592 Å in the salicylic acid molecule, in which the plane of the carboxyl group is tilted by 9° with respect to the plane of the six-membered ring. In the 2,5-piperazinedione molecule the six-membered ring is nearly planar. Bond lengths and angles are generally as expected.

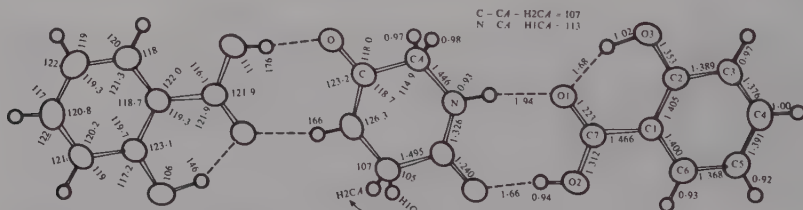


Fig. 1. Bond lengths and angles in the 1:2 complex of 2,5-piperazinedione and salicylic acid; dashed lines indicate hydrogen bonds.

N-ETHYL-N-METHYLMORPHOLINIUM 7,7,8,8-TETRACYANOQUINODIMETHANE (1:2 Complex)



H. KOBAYASHI, 1982. Bull. Chem. Soc. Jpn., 55, 2693-2696.

Triclinic, P1̄, a = 7.796, b = 15.308, c = 6.993 Å, α = 111.96, β = 74.87, γ = 111.82°, Z = 1. Mo radiation, R = 0.088 for 1710 reflexions (at 325 K).

The structure is essentially identical to the structure at 113K and at 323 K (1). Details are in Fig. 1.

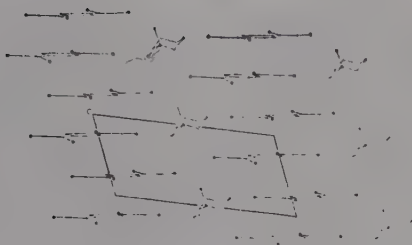


Fig. 1. The crystal structure of  $C_7H_6NO^+$ ,  $C_{12}H_4N_4^-$ .

1. Structure Reports, 43B, 792; 48B, 754.

1-METHYL-3-PROPYLIMIDAZOLIUM 7,7,8,8-TETRACYANO-p-QUINODIMETHANE

$C_{19}H_{17}N_6$

$C_7H_{13}N_2^+$ ,  $C_{12}H_4N_4^-$

V. LANGER, K. HUML and G. RECK, 1982. Acta Cryst., B38, 298-300.

Triclinic,  $P\bar{1}$ ,  $a = 10.982$ ,  $b = 14.337$ ,  $c = 8.750$  Å,  $\alpha = 91.37$ ,  $\beta = 139.09$ ,  $\gamma = 94.40^\circ$ ,  $D_m = 1.211$ ,  $Z = 2$ . Cu radiation,  $R = 0.042$  for 2464 reflexions.

Bond lengths and angles are shown in Fig. 1. The tetracyanoquinodimethane anion is significantly nonplanar whereas the imidazole ring can be considered planar. The interplanar distance between tetracyanoquinodimethane anions is 3.13 Å.

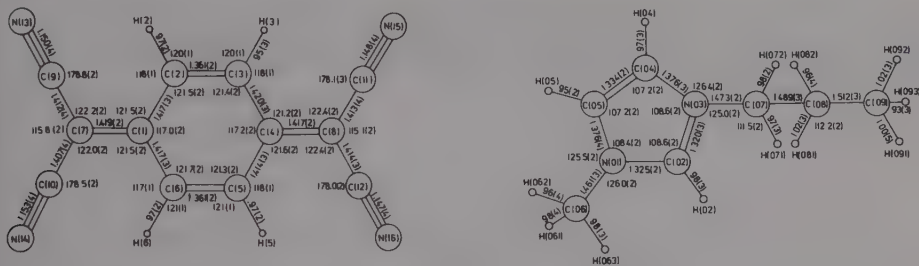


Fig. 1. Bond lengths and angles in the tetracyanoquinodimethane anion and the methylpropylimidazolium cation.

BIS(TETRAMETHYLTETRASELENAFULVALENE) DIHYDROGENTRIFLUORIDE

$C_{20}H_{26}F_3Se_8$

$(C_{10}H_{12}Se_4)_2H_2F_3$

M.A. BENO, G.S. BLACKMAN, J.M. WILLIAMS and K. BECHGAARD, 1982. Inorg. Chem., 21, 3860-3862.

Triclinic,  $P\bar{1}$ ,  $a = 7.209$ ,  $b = 7.691$ ,  $c = 12.837$  Å,  $\alpha = 90.63$ ,  $\beta = 85.69$ ,  $\gamma = 71.15^\circ$ ,  $Z = 1$ . Mo radiation,  $R = 0.070$  for 3099 reflexions at 298 K.  $a = 7.068$ ,  $b = 7.657$ ,  $c = 12.753$  Å,  $\alpha = 90.54$ ,  $\beta = 85.69$ ,  $\gamma = 70.91^\circ$ . Mo radiation,  $R = 0.050$  for 2304 reflexions at 125 K.

The structure (Fig. 1) contains  $H_2F_3^-$  anions disordered about centres of symmetry. The anions cause the columns of the  $C_{10}H_{12}Se_4$  stacks to form segregated sheets in the  $ab$  plane. The stacks are not dimerized.

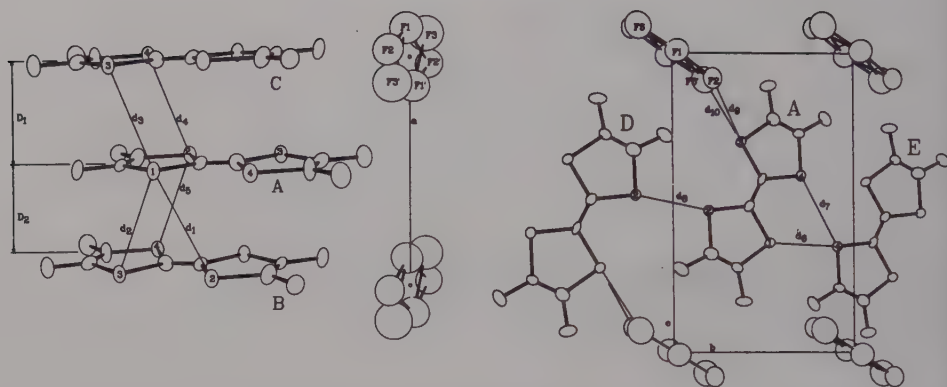
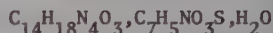
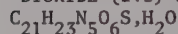


Fig. 1. Two views of the structure of  $(C_{10}H_{12}Se_4)_2H_2F_3$ .

2,4-DIAMINO-5-(3,4,5-TRIMETHOXYBENZYL)PYRIMIDINE 1,2-BENZISOTHAZOL-3(2H)-ONE 1,1-DIOXIDE (1:1) MONOHYDRATE



N. SHIMIZU and S. NISHIGAKI, 1982. *Acta Cryst.*, B38, 1834-1836.

Triclinic,  $P\bar{1}$ ,  $a = 9.152$ ,  $b = 11.487$ ,  $c = 12.674$  Å,  $\alpha = 89.81$ ,  $\beta = 103.97$ ,  $\gamma = 113.95^\circ$ ,  $D_m = 1.397$ ,  $Z = 2$ . Mo radiation,  $R = 0.082$  for 4486 reflexions.

The molecules (Fig. 1) are extensively linked by hydrogen bonds. Molecular geometry (i.e.  $N(1)-C(1)$  1.357(4),  $N(1)-C(4)$  1.376(6),  $S-N(5)$  1.618(5) Å and  $C(1)-N(1)-C(4)$  120.8(4),  $S-N(5)-C(21)$  111.2°) indicates protonation at  $N(1)$  with the proton released from  $N(5)$ . The  $C(2)-C(3)-C(5)-C(6)$  and  $C(3)-C(5)-C(6)-C(7)$  torsion angles are  $-67.5(4)$  and  $122.1(4)^\circ$  respectively.

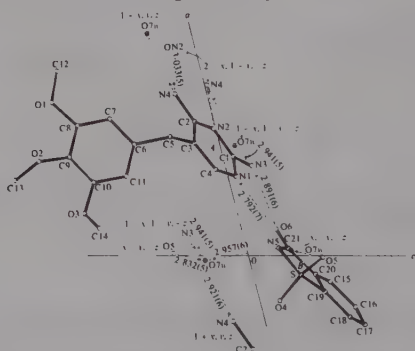
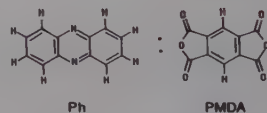


Fig. 1. Intermolecular hydrogen bonds in  $C_{21}H_{23}N_5O_6S \cdot H_2O$ .

PHENAZINE PYROMELLITIC DIANHYDRIDE (1:1 COMPLEX)



N. KARL, W. KETTERER and J.J. STEZOWSKI, 1982. *Acta Cryst.*, B38, 2917-2919.





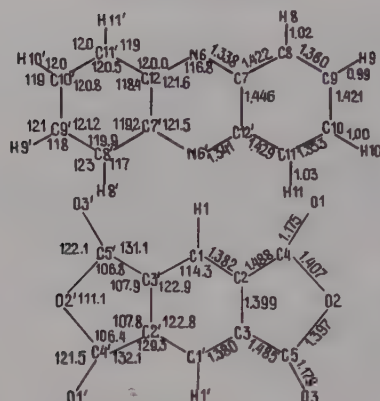
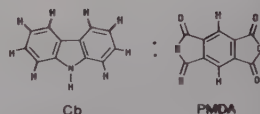
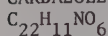


Fig. 1. Distances and angles in  $C_{12}H_9N:C_{10}H_2O_6$ .

# CARBAZOLE PYROMELLITIC DIANHYDRIDE (1:1 COMPLEX)



J.J. STEZOWSKI, W. BINDER and N. KARL, 1982. *Acta Cryst.*, B38, 2912-2914.

Monoclinic,  $P2_1/c$ ,  $a = 6.9778$ ,  $b = 9.301$ ,  $c = 13.366$  Å,  $\beta = 104.679^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.045$  for 3037 reflexions (at  $\sim 120$  K).

The donor carbazole molecules and acceptor pyromellitic dianhydride molecules (Fig. 1) are required by crystallographic symmetry to lie on inversion centres; consequently the non-centrosymmetric carbazole molecules are dipolarly disordered, but little trouble was encountered in resolving the disorder. Similar disorder occurs in the acridine-pyromellitic dianhydride complex (1). The crystal packing consists of infinite stacks of alternating planar donor and acceptor molecules with interplanar spacing 3.30(1) Å and interplanar angle  $2.9(1)^\circ$ .

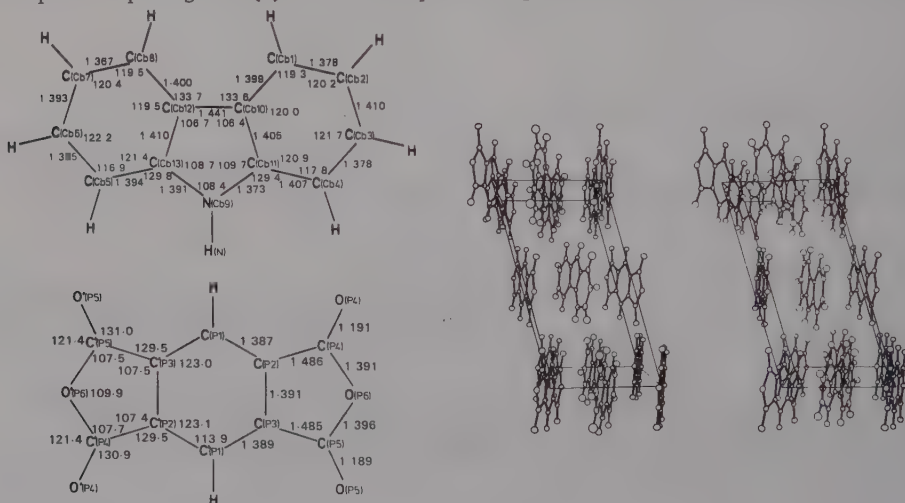


Fig. 1.  $C_{12}H_9N:C_{10}H_2O_6$ : bond lengths and angles in the donor carbazole molecule (upper left) and the acceptor pyromellitic dianhydride molecule (lower left) and a stereoscopic projection on the ac plane displaying crystal packing (right).

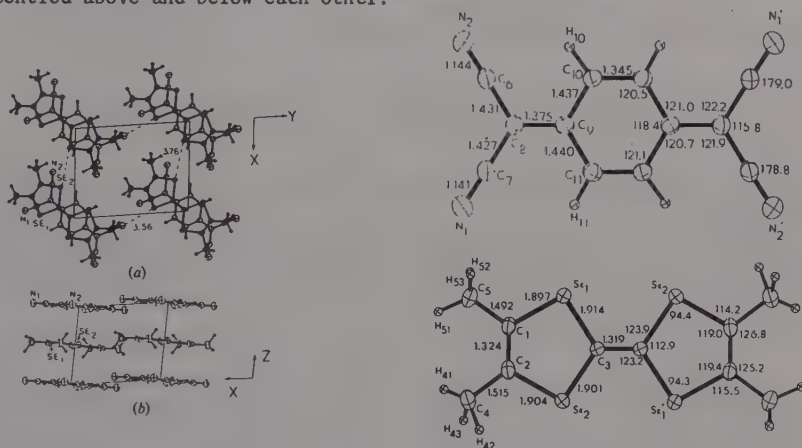
1. This volume, page 718.

4,4',5,5'-TETRAMETHYL- $\Delta^2,2'$ -BI-1,3-DISELENOLE 7,7,8,8-TETRACYANO-p-QUINODIMETHANE  
 $C_{22}H_{16}N_4Se_4$   $C_{10}H_{12}Se_4 \cdot C_{12}H_4N_4$

T.J. KISTENMACHER, T.J. EMGE, A.N. BLOCH and D.O. COWAN, 1982. Acta Cryst., B38, 1193-1199.

Triclinic,  $P\bar{1}$ ,  $a = 8.096$ ,  $b = 10.465$ ,  $c = 6.998$  Å,  $\alpha = 103.78$ ,  $\beta = 98.49$ ,  $\gamma = 94.91^\circ$ ,  $D_m = 1.94$ ,  $Z = 1$ . Mo radiation,  $R = 0.045$  for 2306 reflexions.

The structure (Fig. 1) of this charge-transfer complex, and, semiconducting form (TMTSF-TCNQ), is composed of stacks of alternating TMTSF and TCNQ molecules positioned about centres of symmetry along the crystallographic  $c$  axis and separated by  $\sim 3.5$  Å. The donor TMTSF and acceptor TCNQ molecules are each planar, and are directly centred above and below each other.



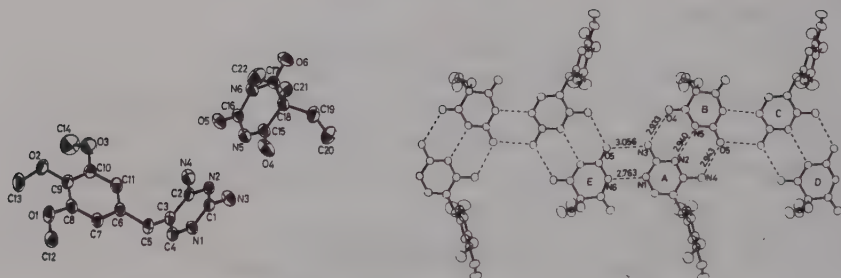
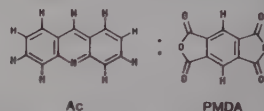


Fig. 1.  $C_{14}H_{16}N_4O_3 \cdot C_8H_{12}N_2O_3$ : molecular structure and packing diagram; broken lines indicate hydrogen bonds.

# ACRIDINE PYROMELLITIC DIANHYDRIDE (1:1 COMPLEX)

$C_{23}H_{11}NO_6$



W. BINDER, N. KARL and J.J. STEZOWSKI, 1982. *Acta Cryst.*, **B38**, 2915-2916.

Triclinic,  $P\bar{1}$ ,  $a = 7.092$ ,  $b = 10.886$ ,  $c = 7.111$  Å,  $\alpha = 117.31$ ,  $\beta = 110.69$ ,  $\gamma = 98.36^\circ$ ,  $Z = 1$ . Mo radiation,  $R = 0.050$  for 4558 reflexions (at  $\sim 120$  K).

The donor acridine and acceptor pyromellitic dianhydride molecules (Fig. 1) are required by crystallographic symmetry to lie on inversion centres; consequently the acridine molecule is disordered. Similar disorder occurs in the carbazole pyromellitic dianhydride complex (1).

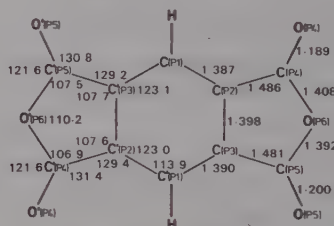
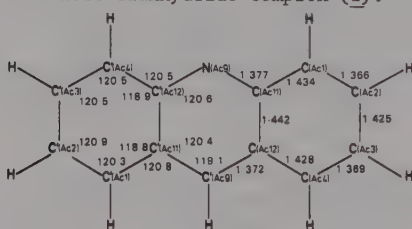


Fig. 1.  $C_{13}H_9N, C_{10}H_2O_6$ : bond lengths and angles in the donor acridine molecule and the acceptor pyromellitic dianhydride molecule.

1. This volume, page 716.

# ADENINE : PHTHALIC ACID 3:1 COMPLEX HEXAHYDRATE

$C_{23}H_{33}N_5O_{10}$

$3(C_5H_5N_5) \cdot C_8H_6O_4 \cdot 6H_2O$

A. TAKENAKA and Y. SASADA, 1982. *Bull. Chem. Soc. Jpn.*, **55**, 680-686.

Triclinic,  $P\bar{1}$ ,  $a = 14.232$ ,  $b = 16.730$ ,  $c = 7.336$  Å,  $\alpha = 78.97$ ,  $\beta = 96.14$ ,  $\gamma = 68.99^\circ$ ,  $D_m = 1.437$ ,  $Z = 2$ . Mo radiation,  $R = 0.085$  for 4076 reflexions.

The two carboxyl groups of the phthalic acid are ionized and they are hydrogen bonded to the amino group and protonated N(1) of two adeninium cations. The third adenine molecule is neutral and disordered. The molecules are linked by an extensive hydrogen bond network which includes a C-H...N hydrogen bond (C...N 3.221(6) Å).

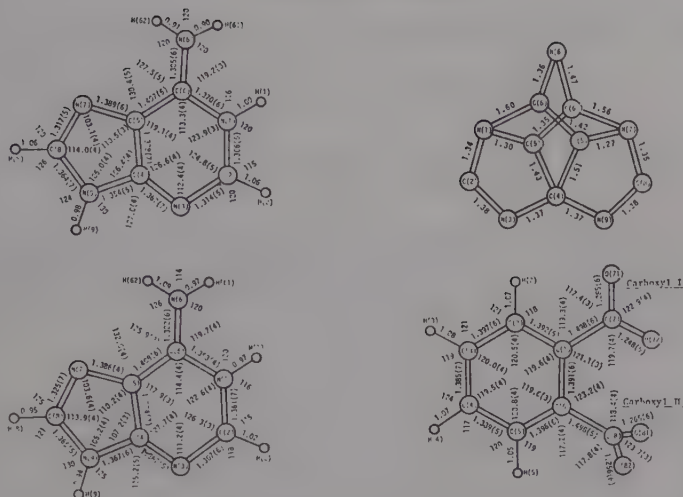


Fig. 1. Dimensions for the phthalic acid and three adenine molecules.

DIBENZOTETRATHIAFULVALENE 2,3,5,6-TETRAFLUORO-7,7,8,8-TETRACYANOQUINODIMETHANIDE  
 $C_{26}H_8F_4N_4S_4$  (DBTTF:TCNQF4)

T.J. EMGE, W.A. BRYDEN, F.M. WIYGUL, D.O. COWAN and T.J. KISTENMACHER, 1982. J. Chem. Phys., **77**, 3188-3197.

Triclinic,  $P\bar{1}$ ,  $a = 13.159$ ,  $b = 13.703$ ,  $c = 7.533$  Å,  $\alpha = 102.89$ ,  $\beta = 105.98$ ,  $\gamma = 63.47^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.063$  for 2797 reflexions.

The structure consists of segregated stacks of donor and acceptor molecules. Within both stacks, significant dimerization is observed. Plane-plane separations are 3.35 and 3.68 Å for DBTTF, and 3.18 and 3.54 Å for TCNQF4. From the geometries of the ions (Fig. 1) it is concluded that charge transfer is complete.

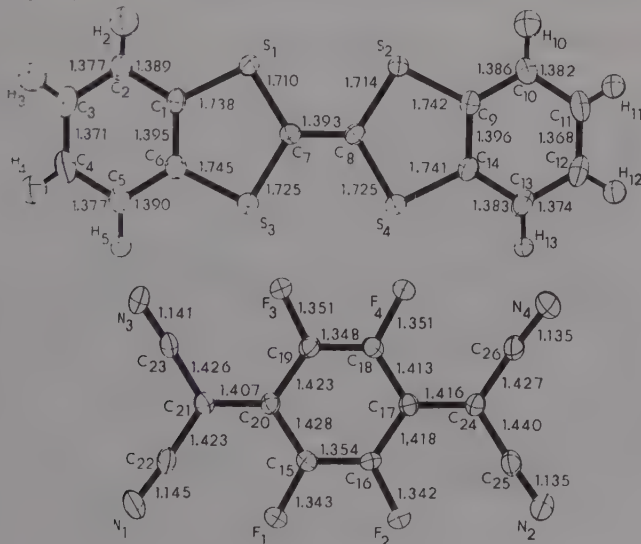


Fig. 1. Dimensions for the DBTTF and TCNQF4 moieties.





5-(1-n-BUTYL)PHENAZINIUM 2,3,5,6-TETRAFLUORO-7,7,8,8-TETRACYANOQUINODIMETHANIDE  
 $C_{28}H_{17}F_4N_6$   $NBP^+$ ,  $TCNQF_4^-$

R.M. METZGER, N.E. HEIMER, D. GUNDEL, H. SIXL, R.H. HARMS, H.J. KELLER, D. NOTHE and D. WEHE, 1982. J. Chem. Phys., **77**, 6203-6214.

Monoclinic,  $P2_1/c$ ,  $a = 10.972$ ,  $b = 17.557$ ,  $c = 13.523$  Å,  $\beta = 111.88^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.056$  for 2463 reflexions.

The structure consists of  $(NBP^+)_2$  and  $(TCNQF_4^-)_2$  dimers stacked in a DDAA sequence along the  $c$  axis. The  $NBP^+$  and  $TCNQF_4^-$  ions are planar, with interplanar distances of 3.54(2) Å for a donor pair and 3.15(3) Å for an acceptor pair. The angle between the  $NBP^+$  and  $TCNQF_4^-$  planes is  $15.8^\circ$ . Dimensions for the  $NBP^+$  and  $TCNQF_4^-$  ions are in Fig. 1.

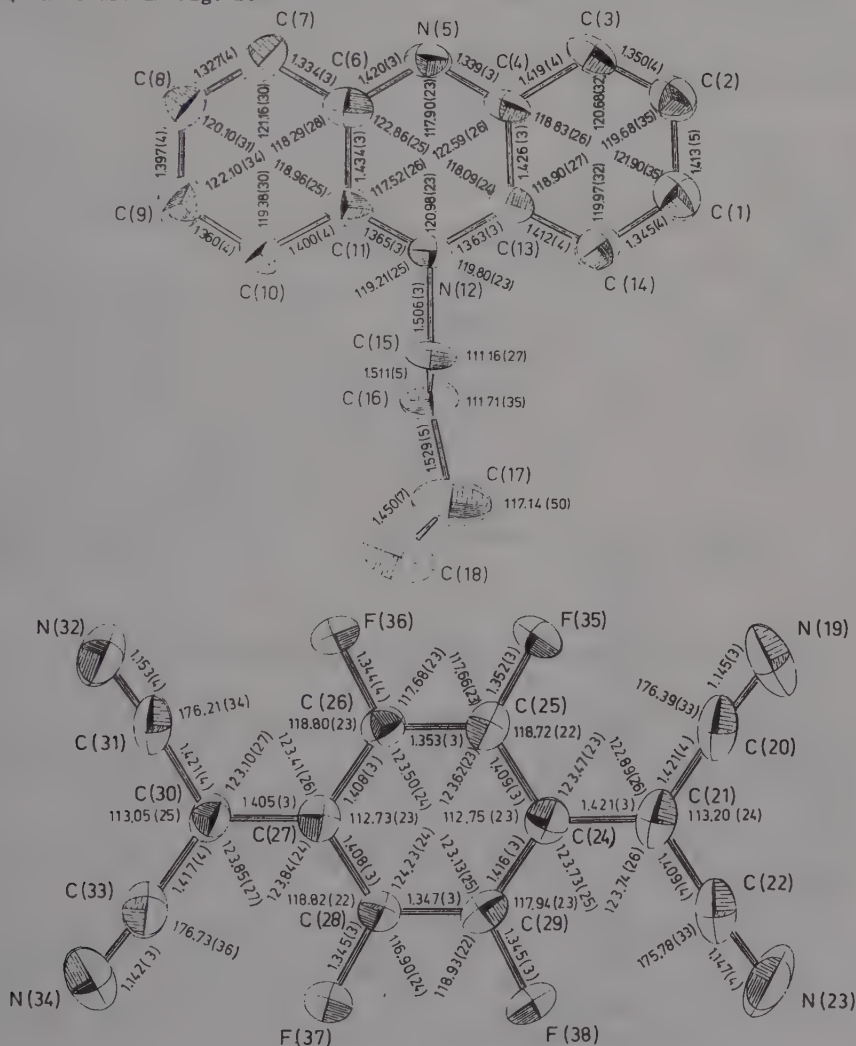
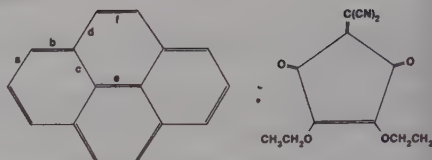
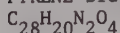


Fig. 1. The  $NBP^+$  and  $TCNQF_4^-$  ions and dimensions.



## PYRENE DICYANOMETHYLENECROCONATE



R.M. DOHERTY, J.M. STEWART, A.D. MIGHELL, C.R. HUBBARD and A.J. FATIADI, 1982. *Acta Cryst.*, **B38**, 859-863.

Triclinic,  $P\bar{1}$ ,  $a = 10.085$ ,  $b = 10.646$ ,  $c = 11.037$  Å,  $\alpha = 98.73$ ,  $\beta = 92.61$ ,  $\gamma = 107.36^\circ$ ,  $D_m = 1.35$ ,  $Z = 2$ . Cu radiation,  $R = 0.048$  for 2767 reflexions!

The unit cell of this charge-transfer complex consists of two crystallographically independent pyrene molecules, each on a centre of symmetry, and two dicyanomethylenecroconate molecules (Fig. 1). Bond distances and angles in the donor pyrene molecule agree with reported values, as do those in the acceptor dicyanomethylenecroconate molecule. The complex consists of stacks of alternating donor and acceptor molecules.

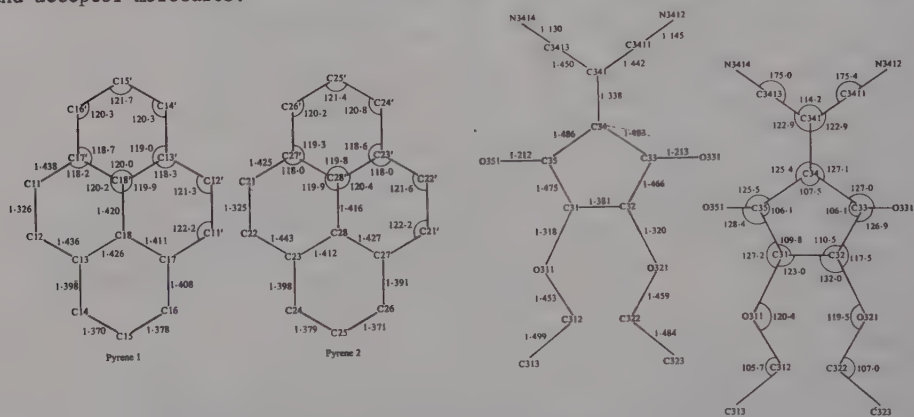
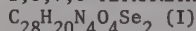


Fig. 1.  $\text{C}_{28}\text{H}_{20}\text{N}_2\text{O}_4$ : bond distances and angles in the pyrene and dicyanomethylenecroconate molecules.

## 2,3,7,8-TETRAMETHOXYSELENANTHRENE 7,7,8,8-TETRACYANOQUINODIMETHANE



## 2,3,7,8-TETRAMETHOXYTHIANTHRENE 7,7,8,8-TETRACYANOQUINODIMETHANE



W. HINRICHS and G. KLAR, 1982. *J. Chem. Research*, **S**, 336-337; **M**, 3540-3593.

I, II. Monoclinic,  $P2_1/c$ ,  $a = 10.481$ ,  $10.441$ ,  $b = 17.940$ ,  $17.914$ ,  $c = 13.818$ ,  $13.757$  Å,  $\beta = 94.99$ ,  $94.31^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.067$ ,  $0.050$  for 4514, 5069 reflexions.

Complexes I and II (Fig. 1) are isostructural and their crystal structures are built up from neutral component molecules whose structural details correspond to those of the pure compounds. The structures are composed of stacks in which the components alternate. In the stacks, each of the folded selenanthrene or thianthrene molecules is arranged in such a way that one of its phenyl rings is coplanar with the TCNQ molecule. The intermolecular separations are 3.34 and 3.57 in I and 3.00 and 3.58 Å in II.

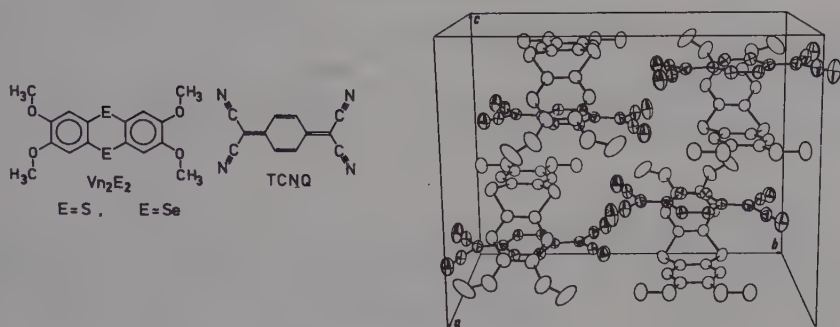


Fig. 1.  $C_{28}H_{20}N_4O_4S_2$ : the molecular skeleton and crystal structure.

5,10-DIHYDRO-5,10-DIETHYLPHENAZINIUM TETRACYANOQUINODIMETHANIDE

$C_{28}H_{22}N_6$

$C_{16}H_{18}N_2^+, C_{12}H_4N_4^-$

K. DIETZ, H. ENDRES, H.J. KELLER, W. MORONI and D. WEHE, 1982. Z. Naturforsch., 37b, 437-442.

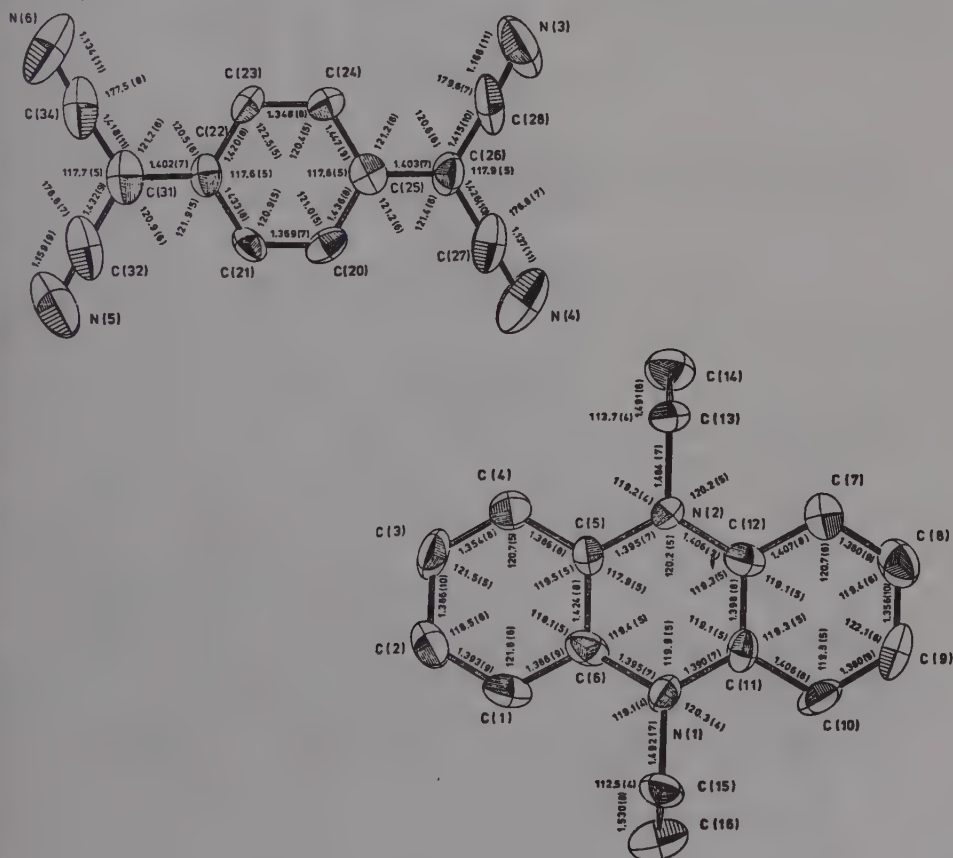


Fig. 1. Bond distances (Å), angles (°), and e.s.d.'s in  $C_{12}H_4N_4^-$ , and in  $C_{16}H_{18}N_2^+$ .

Triclinic,  $P\bar{1}$ ,  $a = 7.739$ ,  $b = 15.899$ ,  $c = 17.037$  Å,  $\alpha = 83.44$ ,  $\beta = 88.34$ ,  $\gamma = 83.19^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.066$  for 1880 reflexions.

The structure consists of planar anions and bent cations (Fig. 1) (dihedral angle  $17.7(6)^\circ$  along the N...N line) arranged in regular mixed stacks along  $c$  (interplanar spacing  $3.10(7)$ ,  $3.99(8)$  Å).

# DECAMETHYLFERROCENE 2,3-DICHLORO-5,6-DICYANOQUINONE

$C_{28}H_{31}Cl_2FeN_2O_2$   $[Fe(C_5(CH_3)_5)_2]^+ \cdot [C_6Cl_2(CN)_2O_2(H)]^-$

E. GEBERT, A.H. REIS, Jr., J.S. MILLER, H. ROMMELMANN, and A.J. EPSTEIN, 1982. J. Am. Chem. Soc., 104, 4403-4410.

Orthorhombic,  $Pbna$ ,  $a = 17.027$ ,  $b = 14.497$ ,  $c = 10.616$  Å,  $Z = 4$ . Mo radiation,  $R = 0.054$  for 2409 reflexions.

The structure (Fig. 1) consists of heterosporic stacks of alternating  $(C_5Me_5)_2Fe^+$  and  $C_6Cl_2(CN)_2O_2(H)^-(DDQH)^-$  ions along the  $c$  axis. Each ion has  $C_2$  symmetry, the latter being disordered. The planar  $DDQH^-$  anion and  $C_5Me_5$  rings of the  $(C_5Me_5)_2Fe^+$  ion are separated by  $3.564$  Å. The angle between the  $C_5$  and  $DDQH^-$  planes is  $3.33^\circ$ . Ring geometries are shown in Fig. 2. The mean  $Fe-C$ ,  $C-C(Cp)$ , and  $C-Me$  distances are  $2.096(2)$ ,  $1.422(3)$ , and  $1.505(3)$  Å. The  $DDQH^-$  geometry is intermediate between quinoid and benzenoid states.

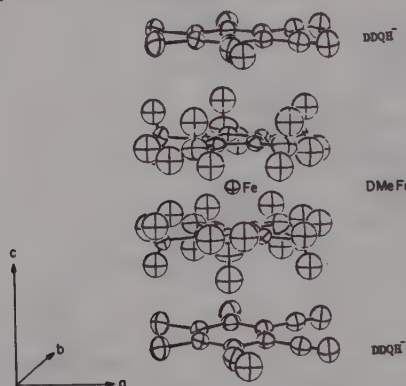
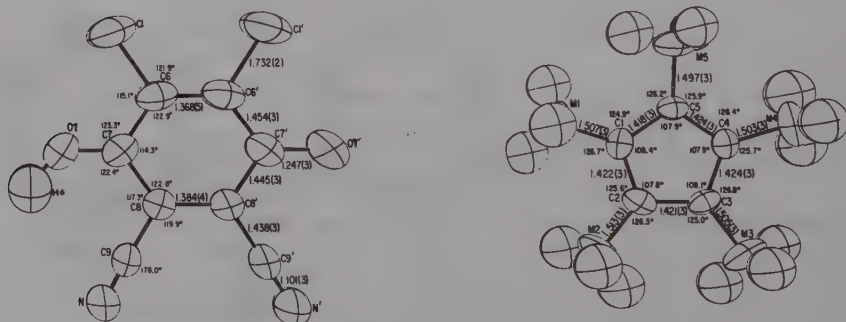
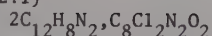
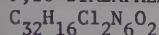


Fig. 1. Stacking of  $DDQH^-$  and  $(C_5Me_5)_2Fe^+$  along  $c$ .



9,10-DIAZAPHENANTHRENE 2,3-DICHLORO-5,6-DICYANO-1,4-BENZOQUINONE (2:1)



B. SHAANAN, U. SHMUELI and M. COLAPIETRO, 1982. Acta Cryst., B38, 818-824.

Monoclinic,  $P2_1/a$ ,  $a = 13.022$ ,  $b = 11.521$ ,  $c = 18.386$  Å,  $\beta = 97.36^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.045$  for 1738 reflexions.

The structure (Fig. 1) consists of sheets of 'dimerized' donors (diazaphenanthrene) which alternate with relatively flat layers of the acceptor (dichlorodicyanobenzoquinone) molecules. Significant attractive donor-acceptor interaction is suggested by short contacts (2.85-2.92 Å) between the N atoms of the donors and the quinoid ring of the acceptor. The charge-transfer interaction is probably of the  $n-\pi^*$  type.

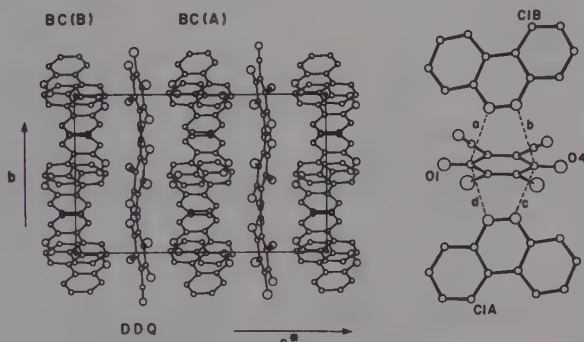
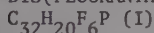
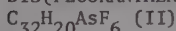


Fig. 1.  $2\text{C}_{12}\text{H}_8\text{N}_2, \text{C}_8\text{Cl}_2\text{N}_2\text{O}_2$ : projections of the structure down the  $a$  axis (left) and down a direction close to the plane normals of diazaphenanthrene molecules A and B (right).

BIS(FLUORANTHENE) HEXAFLUOROPHOSPHATE



BIS(FLUORANTHENE) HEXAFLUOROARSENATE



V. ENKELMANN, B.S. MORRA, Ch. KROHNKE, G. WEGNER and J. HEINZE, 1982. Chem. Phys., 66, 303-313.

I. Monoclinic,  $A2/m$ ,  $a = 6.61$ ,  $b = 12.57$ ,  $c = 14.77$  Å,  $\beta = 104.0^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.10$  for 639 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 6.50$ ,  $b = 12.49$ ,  $c = 14.75$  Å,  $\beta = 104.0^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.08$  for 724 reflexions (at 110 K).

In I, the fluoranthene molecules lie across mirror planes and the anions are on sites of  $2/m$  symmetry. In II, the fluoranthene molecule is in a general location, with the  $\text{AsF}_6^-$  anion on a site of inversion symmetry. Plane-to-plane separations are 3.28 and 3.33 Å in I and 3.22 and 3.28 Å in II. In both complexes (Fig. 1) the stacks of aromatic rings are arranged to leave channels in which the anions are located. Bond lengths and angles in the fluoranthene molecules do not differ significantly in the two structures.

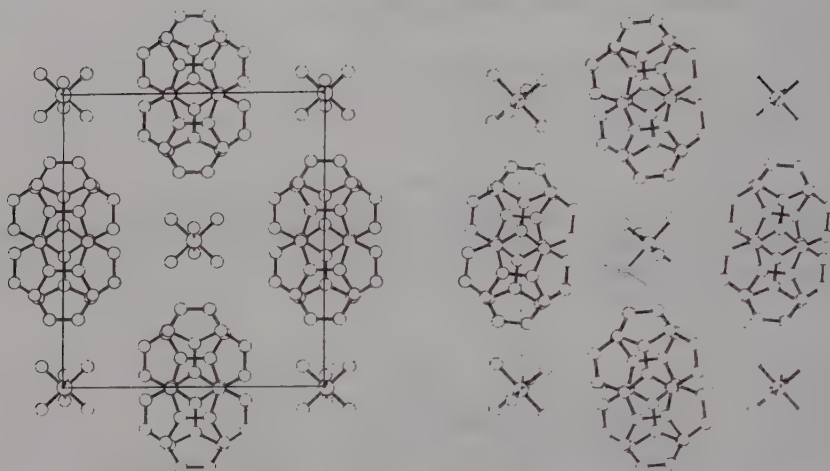


Fig. 1. Projections of the bis(fluoranthene) complexes on the bc plane ( $\text{PF}_6^-$  at left,  $\text{AsF}_6^-$  at right).

# HYDRATED 2:1 COMPLEX OF ADENOSINE AND PROFLAVINE SULPHATE

$\text{C}_{33}\text{H}_{53}\text{N}_{13}\text{O}_{20.5}\text{S}_{1.5}$

$2\text{C}_{10}\text{H}_{14}\text{N}_5\text{O}_4^+, \text{C}_{13}\text{H}_{12}\text{N}_3^+, 1.5\text{SO}_4^{2-}, 6.5\text{H}_2\text{O}$

P. SWAMINATHAN, E. WESTHOF and M. SUNDARALINGAM, 1982. *Acta Cryst.*, B38, 515-522.

Monoclinic,  $\text{P}2_1$ ,  $a = 15.166$ ,  $b = 6.715$ ,  $c = 22.384 \text{ \AA}$ ,  $\beta = 96.4^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.14$  for 2897 reflexions.

The two adenosine molecules in the asymmetric unit are base-paired through  $\text{N}(6)\cdots\text{N}(7)$  hydrogen bonds. Proflavine is sandwiched between these pairs at a distance of  $3.36 \text{ \AA}$  along  $b$  (Fig. 1). The complex exhibits extensive stacking and is additionally stabilised by hydrogen bonds involving proflavine, adenosine and sulphate ions. One amino proton of proflavine is involved in a bifurcated hydrogen bond to the ribose hydroxyl  $\text{O}(2')\text{A}$  and a furanose ring oxygen atom  $\text{O}(4')\text{A}$ , (Fig. 1). The structure is disordered with one  $\text{SO}_4^{2-}$  ion sharing its site with three water molecules. The fully occupied  $\text{SO}_4^{2-}$  forms a hydrogen-bonded bridge to the quaternary nitrogen of proflavine and the amino group of adenine B stacked over it. There is a puckering disorder in ribose B interpreted as a 4:1 mixture of two conformers  ${}^0\text{E}[\text{O}(4')\text{-endo}]$  and  ${}^2\text{E}[\text{C}(2')\text{-endo}]$ . The ordered ribose of adenosine A exhibits flattened symmetrical twist conformation,  ${}^3\text{T}[\text{P} = 179(5)^\circ, \tau_m = 24(1)^\circ]$ , with an intramolecular hydrogen bond of  $2.63(2) \text{ \AA}$  between the ribose hydroxyls.

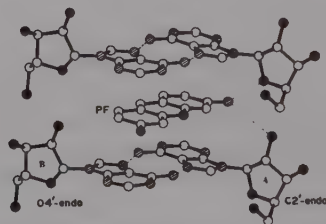
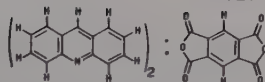
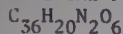


Fig. 1. Edge-on view of the adenosine-proflavine complex.



## ACRIDINE PYROMELLITIC DIANHYDRIDE (2:1 COMPLEX)



N. KARL, W. BINDER, P. KOLLAT and J.J. STEZOWSKI, 1982. Acta Cryst., B38, 2919-2921.

Monoclinic,  $P2_1/c$ ,  $a = 9.807$ ,  $b = 7.581$ ,  $c = 17.835$  Å,  $\beta = 100.18^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.062$  for 4946 reflexions (at  $\sim 120$  K).

The bonding geometry of the pyromellitic dianhydride molecule (Fig. 1) is representative of that found for the uncomplexed molecule (1) and for the acceptor in the 1:1 donor-acceptor complexes with donors such as anthracene (2) and phenazine (3). Crystallographic symmetry requires the pyromellitic dianhydride molecule to occupy an inversion centre in the title complex; the acridine molecule is in a general position. The crystals consist of discrete donor-receptor-donor sandwiches.

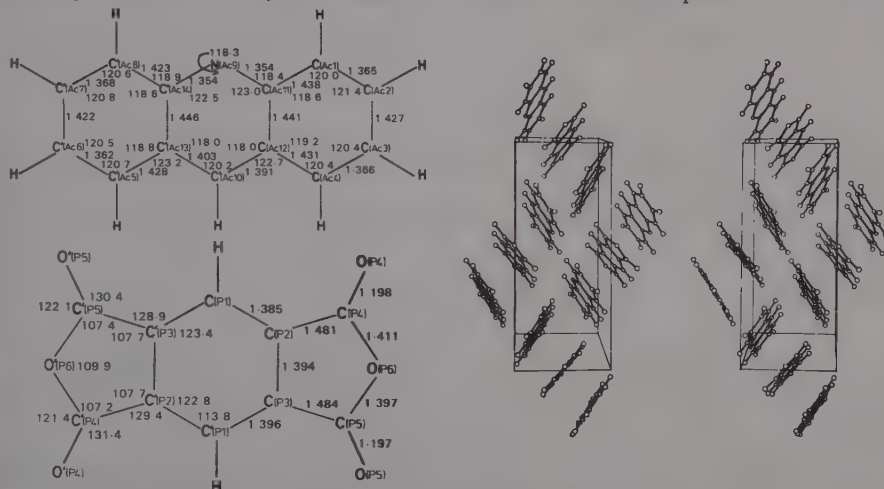


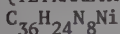
Fig. 1.  $2\text{C}_{13}\text{H}_9\text{N}, \text{C}_{10}\text{H}_2\text{O}_6$ : bond lengths and angles in the donor acridine molecule (upper left) and the acceptor pyromellitic dianhydride molecule (lower left) and a stereoscopic projection on the bc plane displaying one layer of crystal packing (right).

1. Structure Reports, 45B, 320.

2. Ibid, 44B, 563.

3. This volume, page 714.

## (TETRAMETHYLPORPHINATO)NICKEL(II) 7,7,8,8-TETRACYANO-p-QUINODIMETHANE



L.J. PACE, A. ULMAN and J.A. IBERS, 1982. Inorg. Chem., 21, 199-207.

Monoclinic,  $P2_1/n$ ,  $a = 17.542$ ,  $b = 6.868$ ,  $c = 22.597$  Å,  $\beta = 98.42^\circ$ ,  $D_m = 1.50$ ,  $Z = 4$ . Mo radiation,  $R = 0.053$  for 3095 reflexions.

The structure consists of stacks of alternating, parallel  $\text{Ni}(\text{C}_{24}\text{H}_{12}\text{N}_4)$  and  $\text{C}_{12}\text{H}_4\text{N}_4$  molecules which are tilted with respect to the stacking axis. The short intrastack interplanar spacing ( $\sim 3.30$  Å) and IR data indicate a charge-transfer interaction. Molecular geometries are shown in Fig. 1.



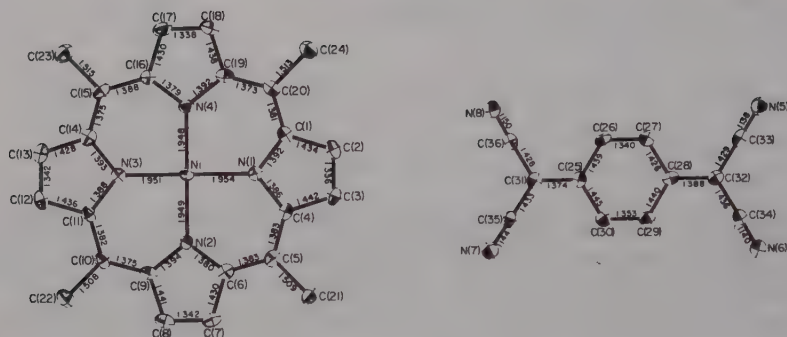


Fig. 1. Bond lengths in  $\text{Ni}(\text{C}_{24}\text{H}_{12}\text{N}_4)$  and  $\text{C}_{12}\text{H}_4\text{N}_4$  in the (1:1) molecular complex.

HEXAMETHYLENETETRASELENAFULVALENE 7,7,8,8-TETRACYANO-p-QUINODIMETHANE 2:1 COMPLEX  
 $\text{C}_{36}\text{H}_{28}\text{N}_4\text{Se}_4$  (HMTSF) $_2$ (TCNQ)

T.J. EMGE, W.A. BRYDEN, D.O. COWAN and T.J. KISTENMACHER, 1982. Mol. Cryst. Liq. Cryst., 90, 173-184.

Monoclinic,  $P2_1/c$ ,  $a = 13.993$ ,  $b = 10.723$ ,  $c = 13.032$  Å,  $\beta = 112.71^\circ$ ,  $D_m = 2.10$ ,  $Z = 2$ . Mo radiation,  $R = 0.066$  for 3315 reflexions.

The TCNQ molecule (Fig. 1) lies on a crystallographic inversion centre and the crystal structure of  $(\text{HMTSF})_2(\text{TCNQ})$  consists of linear chains of alternating donor diads and acceptor monads propagating along the crystallographic  $b$  axis. Within these mixed stacks, the donor/donor overlap is of the ring-over-double bond type (mean separation 3.72 Å), while the donor/acceptor overlap (mean separation 3.46 Å) is such that the HMTSF molecule is very nearly parallel to and directly centered above the adjacent TCNQ molecule. Strong interstack interactions of the types  $\text{Se}(\text{HMTSF})\dots\text{Se}(\text{HMTSF})$ , 3.64 Å, and  $\text{Se}(\text{HMTSF})\dots\text{N}(\text{TCNQ})$ , 3.29 Å, serve to couple adjacent mixed stacks.

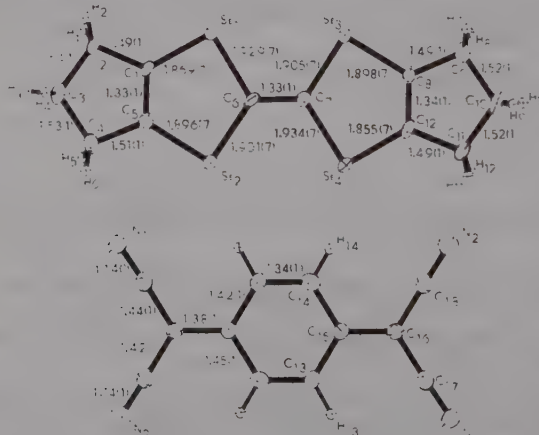
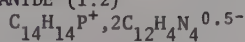
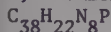


Fig. 1. Bond lengths in the HMTSF and TCNQ molecules.

## 5,5-DIMETHYLDIBENZOPHOSPHOLIUM 7,7,8,8-TETRACYANO-p-QUINODIMETHANIDE (1:2)



G.J. ASHWELL, D.W. ALLEN, D.A. KENNEDY and I.W. NOWELL, 1982. Acta Cryst., B38, 2525-2528.

Monoclinic,  $P2_1/m$ ,  $a = 7.547$ ,  $b = 30.19$ ,  $c = 7.863 \text{ \AA}$ ,  $\beta = 115.86^\circ$ ,  $D_m = 1.30$ ,  $Z = 2$ . Mo radiation,  $R = 0.095$  for 1081 reflexions.

In the crystal structure (Fig. 1) the anions, which adopt a slight boat conformation, stack plane-to-plane in columns parallel to  $c$ , each column consisting of a series of anion dimers. The columns are arranged in sheets parallel to the  $ac$  plane, with successive sheets being interleaved along  $b$  by the cations. Bond distances and angles are generally as expected.

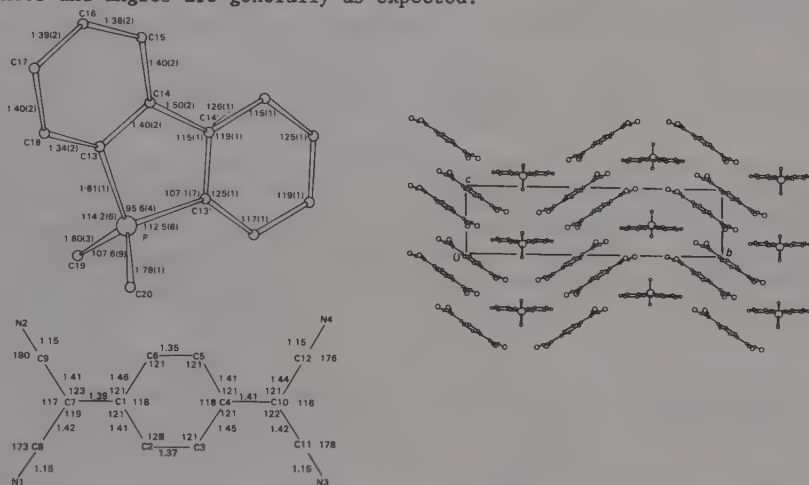
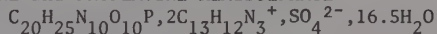
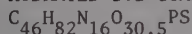


Fig. 1.  $\text{C}_{38}\text{H}_{22}\text{N}_8\text{P}$ : bond distances and angles in the cation (upper left) and anion (lower left) and the projection of the structure along  $a^*$ .

## HYDRATED 1:2 COMPLEX OF ADENYL(3'-5')ADENOSINE and PROFLAVINE HEMISULPHATE



H.-S. SHIEH, H.M. BERMAN, S. NEIDLE, G. TAYLOR and M. SANDERSON, 1982. Acta Cryst., B38, 523-531.

Orthorhombic,  $P2_12_12_1$ ,  $a = 32.157$ ,  $b = 21.450$ ,  $c = 10.175 \text{ \AA}$ ,  $D_m = 1.32$ ,  $Z = 4$ . Cu radiation,  $R = 0.118$  for 3507 reflexions.

The dinucleoside phosphate backbone is extended with the adenines base-paired by  $\text{N}(6) \dots \text{N}(7)$  hydrogen bonds. Each pair is sandwiched by planar proflavine cations protonated at  $\text{N}(10)$  which also stack with each other  $3.4 \text{ \AA}$  apart (Fig. 1). Infinite columns of heterocyclic stacked planes are linked by the hydrophillic ribose-phosphate groups. Solvent molecules, some disordered, lie in the continuous channels between columns of stacked rings. All but one solvent molecule are hydrogen bonded to each other.  $\text{N}(15)$  of proflavine 1 hydrogen bonds to  $\text{O}(1A)$  of a phosphate group and  $\text{N}(15)$  of proflavine (2) hydrogen bonds to the ribosyl  $\text{O}(1')$ . The sulphate ion is disordered. The two sugars differ; the 5' ribose has a  $\text{C}(1')\text{-exo}, \text{C}(2')\text{-endo}$  conformation with  $\text{P}_5 = 142^\circ$ . The glycosidic torsion angle ( $\chi$ ) is  $-120^\circ$ . The 5'-hydroxyl forms a hydrogen bond with the adenine  $\text{N}(3)$ . The other ribose is  $\text{C}(3')\text{-endo}$  ( $\text{P}_5 = 14^\circ$ ,  $\psi = 71^\circ$ ). The  $\alpha$  angle of the molecule is  $262^\circ$  and  $\epsilon = 171^\circ$  leading to a separation of the base planes of  $6.3 \text{ \AA}$ .

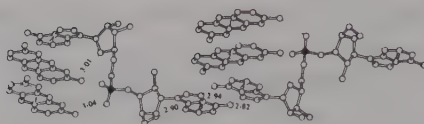


Fig. 1. A view of the ApA-proflavine complex.

$N,N'-(1,3\text{-TRIMETHYLENE})\text{BIS}(2\text{-OXY-1-NAPHTHALDIMEINE})\text{COPPER(II)}\text{ BIS}(7,7,8,8\text{-TETRACYANO-QUINODIMETHANE})$   
 $C_{49}H_{28}CuN_{10}O_2 \cdot CuO_2N_2C_{25}H_{20}, (N_4C_{12}H_4)_2$

N. MATSUMOTO, T. MIYAZAKI, Y. SAGARA and A. OHYOSHI, 1982. *Inorg. Chim. Acta*, **63**, 249-256.

Monoclinic,  $C2/c$ ,  $a = 37.343$ ,  $b = 7.717$ ,  $c = 13.888$  Å,  $\beta = 96.26^\circ$ ,  $D_m = 1.42$ ,  $Z = 4$ .  
 Mo radiation,  $R = 0.069$  for 1263 reflexions.

The coordination about the Cu atom is intermediate between square planar and tetrahedral (Fig. 1) with Cu-O(1) and Cu-N(1) distances of 1.948 and 1.948 Å. The  $\pi$ -electron donors (naphthalene moieties of the Cu complex) and acceptors (TCNQ) stack alternately (Fig. 1) and the naphthalene ring just overlaps one of the two neighbouring TCNQ molecules and partly on the other. The dihedral angles between the planes (overlapped and partly overlapped) are  $8.6$  and  $10.5^\circ$  with the naphthalene ring plane and the intermolecular distances between the TCNQ and naphthalene planes range from 3.27 to 3.50 Å.

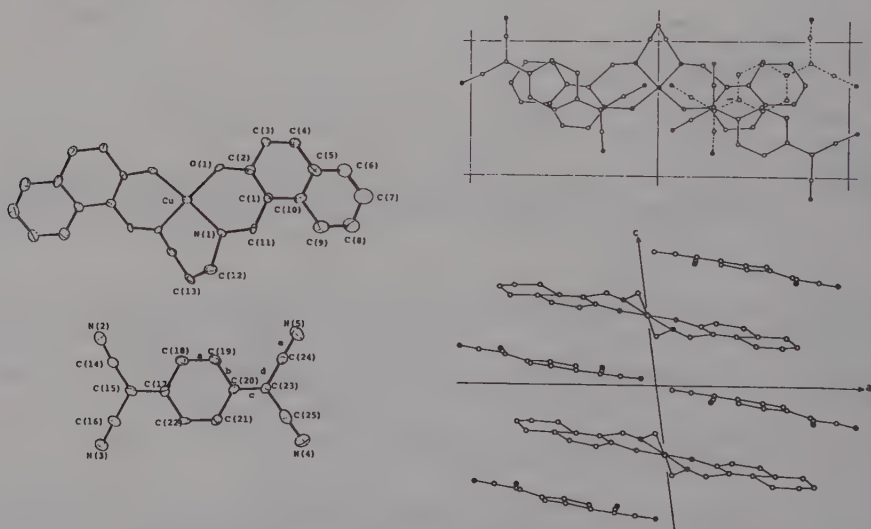


Fig. 1. Molecular structures of  $CuO_2N_2C_{25}H_{20}$  and TCNQ (left), and projections of crystal structures along c-axis (top right) and b-axis (bottom right).

$1,1'\text{-BICOBALTOCENE}[Co^{III}Co^{III}]\text{ TRIS(TETRACYANO-p-QUINODIMETHANIDE)}$   
 $C_{56}H_{28}Co_2N_{12}$

C.-P. LAU, P. SINGH, S.J. CLINE, R. SEIDERS, M. BROOKHART, W.E. MARSH, D.J. HODGSON and W.E. HATFIELD, 1982. *Inorg. Chem.*, **21**, 208-212.

Triclinic,  $P\bar{1}$ ,  $a = 7.475$ ,  $b = 10.113$ ,  $c = 14.306$  Å,  $\alpha = 92.51^\circ$ ,  $\beta = 98.84^\circ$ ,  $\gamma = 92.64^\circ$ ,  $D_m = 1.54$ ,  $Z = 1$ . Mo radiation,  $R = 0.047$  for 1979 reflexions.

The TCNQ units stack to form an alternating chain with two short, adjacent interplanar spacings of 3.15 Å, followed by a longer interplanar spacing of 3.45 Å. The interplanar spacing of the fulvalene moieties in the cation is 3.270 Å and the Co-C bond distances range from 2.012(5) to 2.041(5) Å. Bond lengths and angles are shown in Fig. 1.

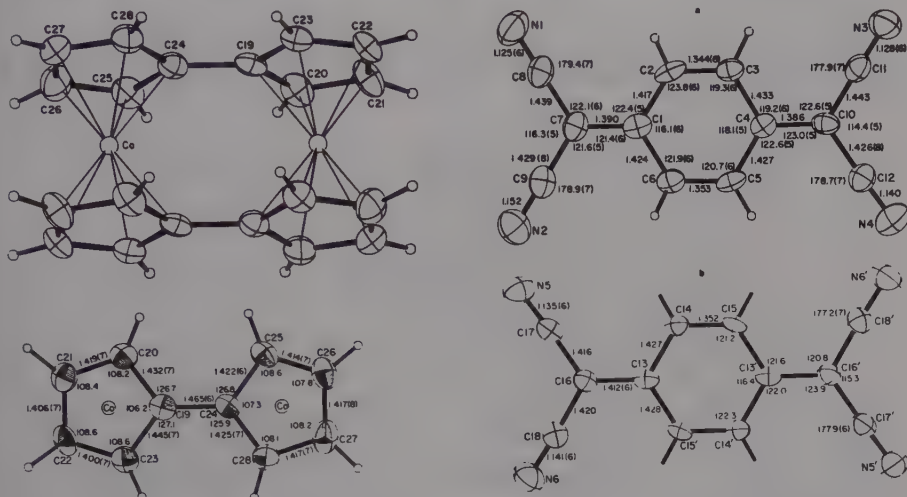
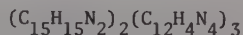
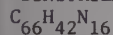


Fig. 1. The 1,1'-bicobaltocene cation (left) and the two independent TCNQ moieties (right).

BIS(5-PROPYLPHENAZINIUM) TRIS[2,2'-(2,5-CYCLOHEXADIENE-1,4-DIYLIDENE) BIS(PROPANEDINITRILE)]



R. HARMS, H.J. KELLER, D. NÖTKE and D. WEHE, 1982. *Acta Cryst.*, B38, 2838-2841.

Triclinic,  $P\bar{1}$ ,  $a = 8.913$ ,  $b = 12.187$ ,  $c = 13.262$  Å,  $\alpha = 96.21$ ,  $\beta = 108.13$ ,  $\gamma = 97.36^\circ$ ,  $Z = 1$ . Mo radiation,  $R = 0.099$  for 1351 reflexions.

In this charge-transfer complex (Fig. 1) all the molecules are planar with the exception of one  $C_{12}H_4N_6$  acceptor molecule, in which the planes through the  $=C(CN)_2$  groups are slightly inclined with respect to the ring plane. Mixed stacks of  $C_{12}H_4N_6$  dimers and  $C_{12}H_4N_6$  trimers occur along the [011] direction.

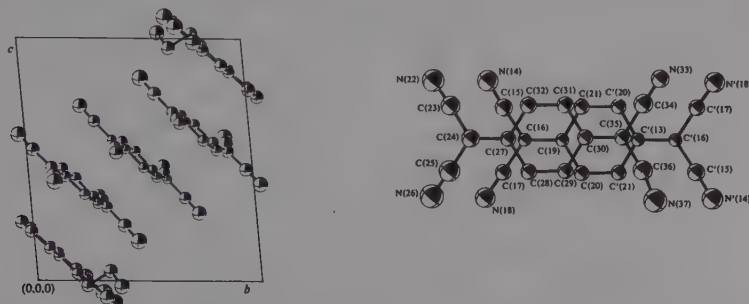


Fig. 1.  $(C_{15}H_{15}N_2)_2(C_{12}H_4N_4)_3$ : projections of the unit cell on to the bc plane and of two adjacent  $C_{12}H_4N_4$  units perpendicular to the ring plane.

TRIS(HYDROQUINONE) SULPHUR DIOXIDE CLATHRATE  
 $C_6H_6O_2 \cdot 0.295(SO_2)$

$3(C_6H_6O_2) \cdot 0.884(SO_2)$

T.M. POLYANSKAYA, V.I. ALEKSEEV, V.V. BAKAKIN and G.N. CHEKHOVA, 1982. Zh. Strukt. Khim., 23-1, 123-127 [J. Struct. Chem., 23, 101-104].

Trigonal,  $R\bar{3}$ ,  $a = 16.31$ ,  $c = 5.821$  Å,  $D_m = 1.46$ ,  $Z = 9$ . Mo radiation,  $R = 0.046$  for 649 reflexions.

The structure (Fig. 1) is that of a type II clathrate of  $\beta$ -hydroquinone. The O...O directions of the disordered  $SO_2$  molecules in the cavities of the framework are not parallel to the  $c$  axis, and weak interactions (S)O...H(O) 2.43 and S...C<sub>6</sub> 3.50 Å are observed.

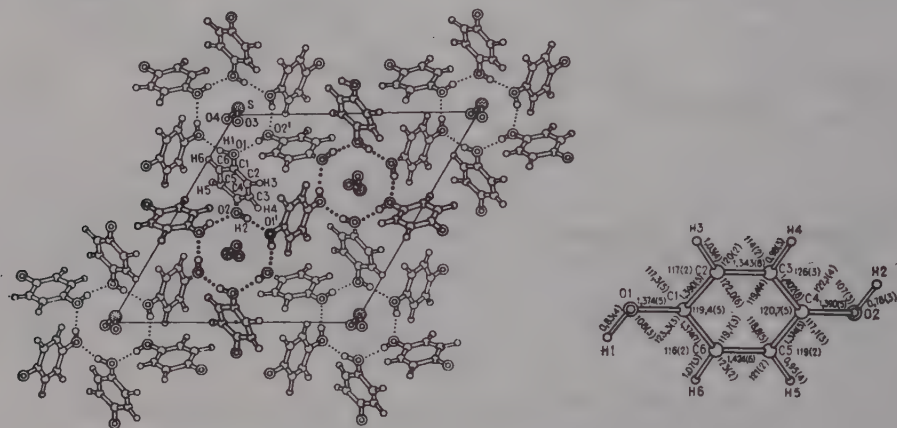


Fig. 1. Structure of  $3(C_6H_6O_2) \cdot 0.884(SO_2)$ , geometry of  $C_6H_6O_2$  at right.

2,2,4,4,6,6-HEXA(1-AZIRIDINYL)CYCLOTRI(PHOSPHAZENE) BENZENE CLATHRATE  
 $C_{12}H_{24}N_9P_3 \cdot 0.5(C_6H_6)$

T.S. CAMERON, J.-F. LABARRE and M. GRAFFEUIL, 1982. Acta Cryst., B38, 168-171.

Rhombohedral,  $R\bar{3}$ ,  $a = 10.266$  Å,  $\alpha = 79.21^\circ$ ,  $Z = 1$ . Mo radiation,  $R = 0.026$  for 1206 reflexions.

The crystal structure (Fig. 1) is made up of two centrosymmetrically related molecules of the cyclotri(phosphazene) and a single molecule of benzene positioned at the bar-three centre. All three molecules contain at least  $C_3$  symmetry. In the cyclotri(phosphazene), the geminal aziridinyl groups adopt an unusual cis conformation; P-N bond distances are 1.590 and 1.597 Å (endocyclic) and 1.670 and 1.677 Å (exocyclic).



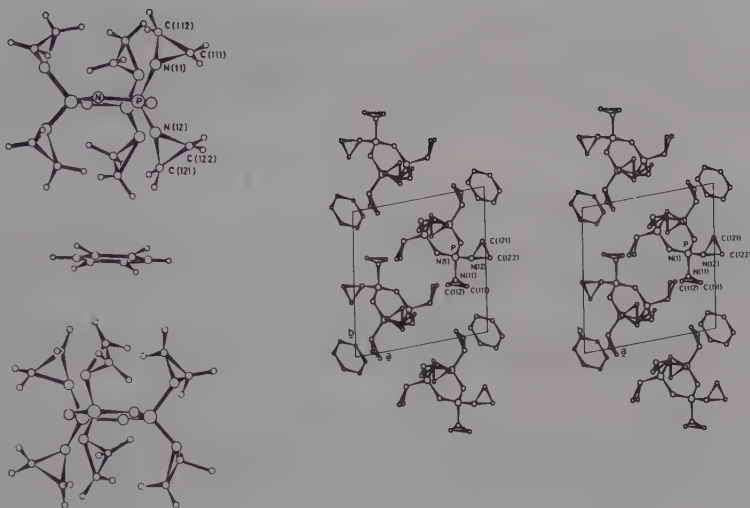


Fig. 1.  $C_{12}H_{24}N_9P_3 \cdot \frac{1}{2}(C_6H_6)$ : view of the molecules almost perpendicular to the  $C_3$  axis and a stereoscopic view of the unit cell along  $c$ .

TRI-*n*-BUTYLPHOSPHINE OXIDE 34.5-WATER CLATHRATE  
 $C_{12}H_{27}OP, 34.5H_2O$

$(n-C_4H_9)_3PO, 34.5H_2O$

V.I. ALEXEEV, Yu.V. GATILOV, T.M. POLYANSKAYA, V.V. BAKAKIN, Yu.A. DYADIN and L.A. GAPONENKO, 1982. Zh. Strukt. Khim., 23-3, 86-91 [J. Struct. Chem., 23, 395-399].

Orthorhombic, Pbam,  $a = 23.479$ ,  $b = 19.949$ ,  $c = 12.136$  Å,  $Z = 4$ . Mo radiation,  $R = 0.143$  for 2553 reflexions (framework structure, actual space group  $P2_12_12_1$ ), (at  $-90^\circ C$ ).

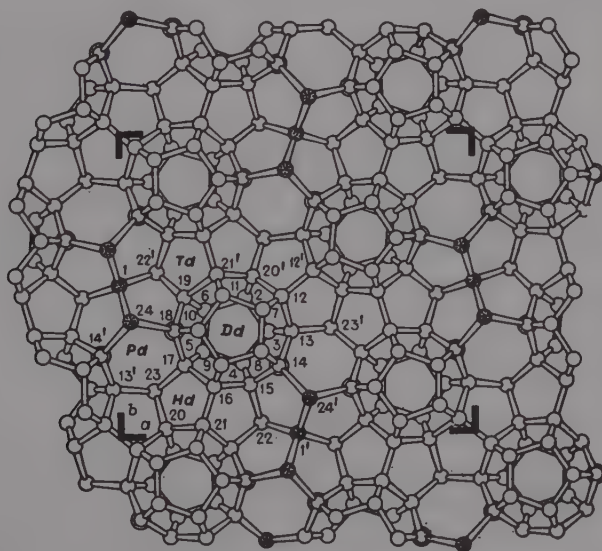


Fig. 1. Projection of the clathrate framework structure in  $(n-C_4H_9)_3PO, 34.5H_2O$ .





III. Monoclinic,  $P2_1/c$ ,  $a = 9.779$ ,  $b = 26.142$ ,  $c = 6.977$  Å,  $\beta = 97.74^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.048$  for 2672 reflexions.

IV. Monoclinic,  $P2_1/c$ ,  $a = 10.016$ ,  $b = 16.742$ ,  $c = 11.241$  Å,  $\beta = 91.68^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.049$  for 2037 reflexions.

V. Monoclinic,  $P2_1/c$ ,  $a = 10.212$ ,  $b = 17.440$ ,  $c = 11.181$  Å,  $\beta = 93.06^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.053$  for 1702 reflexions.

The inclusion complexes of furaltadone hydrochloride with water and acetic acid (IV and V) represent channel-type clathrates in which the guest molecules are accommodated in continuous canals running through the crystal parallel to the  $c$ -axis. The canals have an approximately cylindrical shape and are lined with O and Cl nucleophiles; nevertheless, the guest species are translationally disordered. The structures are compared with those of the furaltadone base (I, Fig. 1) and moxnidazole.HCl (III, Fig. 1). No clathrate-type channels are formed when a hydrate of moxnidazole.HCl crystallizes from water. The structure II features dense packing.

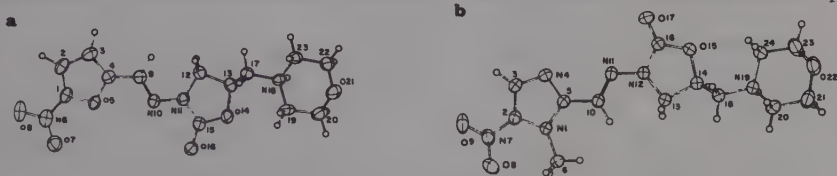
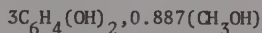


Fig. 1. The (a) furaltadone and (b) moxnidazole moieties (from V and II, respectively).

#### $\beta$ -HYDROQUINONE METHANOL CLATHRATE



T.C.W. MAK, 1982. J. Chem. Soc. Perkin II, 1435-1437.

Rhombohedral,  $R\bar{3}$ ,  $a = 16.621$ ,  $c = 5.562$  Å,  $D_m = 1.343$ ,  $Z = 3$ . Mo radiation,  $R = 0.043$  for 632 reflexions.

The  $C_6O_2$  skeleton of the hydroquinone (Fig. 1) is planar within experimental error, but shows considerable bond angle distortion. In addition to the normal distorting effects of the phenolic hydrogen  $C(3)-C(4)-O(2)$  ( $120.5^\circ$ ,  $C(5)-C(4)-O(2)$  ( $118.1^\circ$ ),  $C(6)-C(1)-O(1)$  ( $125.1^\circ$ ) is considerably enlarged compared with  $C(3)-C(4)-O(2)$  ( $120.5^\circ$ ) and  $C(2)-C(1)-C(6)$  ( $118.3^\circ$ ) is significantly smaller than  $C(3)-C(4)-C(5)$  ( $121.3^\circ$ ), resulting in  $C(1)-O(1)$  making an angle of  $4.3^\circ$  with the long molecular axis through  $C(1), C(4), O(2)$ . These distortions result from the closer contacts between the guest methanol molecule and  $C(6)$  ( $3.71-3.75$ ) compared with  $C(3)$  ( $3.79-3.85$  Å). The methanol has three preferred orientations related by the  $C_3$  symmetry axis along  $c$ , in each of which the  $C(7)-O(3)$  bond is tilted  $35^\circ$  from the  $c$  axis such that  $O(3)$  is brought into closer contact with the basal ring composed of six phenolic hydroxyl groups. The resulting structure can not be described simply by conventional hydrogen bonding schemes.

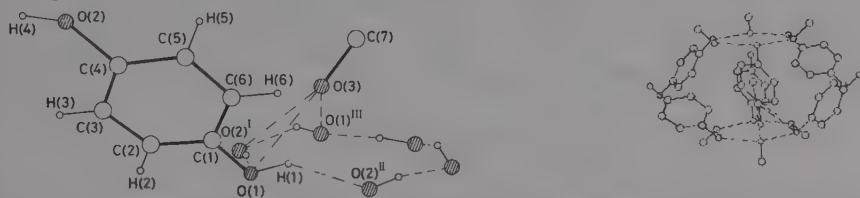
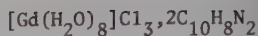


Fig. 1. Molecular structure and host guest structure of  $C_{18}H_{18}O_6, 0.887(CH_4O)$ .

OCTAAQUAGADOLINIUM(III) CHLORIDE 4,4'-BIPYRIDYL CLATHRATE  
 $C_{20}H_{32}Cl_3GdN_4O_8$



M. BUKOWSKA-STRYŻEWSKA and A. TOSIK, 1982. *Acta Cryst.*, **B38**, 265-267.

Tetragonal,  $P\bar{4}c2$ ,  $a = b = 8.901$ ,  $c = 19.319$  Å,  $D_m = 1.53$ ,  $Z = 2$ . Cu radiation,  $R = 0.068$  for 1350 reflexions.

In the clathrate (Fig. 1) the  $[Gd(H_2O)_8]^{3+}$  coordination polyhedron is in the form of an  $S_4$  (4) dodecahedron with Gd-O(1) 2.451 Å and Gd-O(2) 2.354 Å. Chloride ions and 4,4'-bipyridyl molecules are in bridging positions between complex cations, forming O-H...N and O-H...Cl hydrogen bonds involving all the water H atoms. Deformation of the 4,4'-bipyridyl molecular geometry occurs.

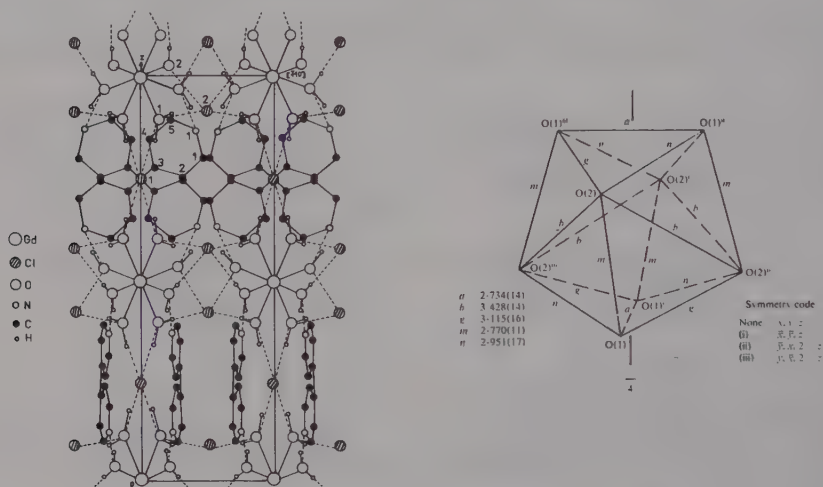


Fig. 1.  $C_{20}H_{32}Cl_3GdN_4O_8$ : view of the unit cell contents along  $[110]$ , and of the  $[Gd(H_2O)_8]^{3+}$  coordination polyhedron showing the lengths of the edges.

BIS(4,4'-BIPYRIDYL) OCTAAQUAYTTRIUM(III) CHLORIDE  
 $C_{20}H_{32}Cl_3Y_3N_4O_8$



M. BUKOWSKA-STRZYŻEWSKA and A. TOSIK, 1982. *Acta Cryst.*, **B38**, 950-951.

Orthorhombic,  $Pcc2$ ,  $a = 8.883$ ,  $b = 8.883$ ,  $c = 19.231$  Å,  $D_m = 1.44$ ,  $Z = 2$ . Mo radiation,  $R = 0.068$  for 1111 reflexions.

The structure (Fig. 1) contains separate  $[Y(H_2O)_8]^{3+}$  ions with Y coordinated to eight water molecules with two different Y-O bond lengths (2.425 and 2.327 Å respectively). The coordination polyhedron forms a  $C_2$  dodecahedron (approximate to  $S_4$ ) connected with the  $Cl^-$  ions by twelve O-H...Cl hydrogen bonds and with the 4,4'-bipyridyl molecules by four O-H...N bonds. This compound is isostructural with the corresponding gadolinium clathrate (1).

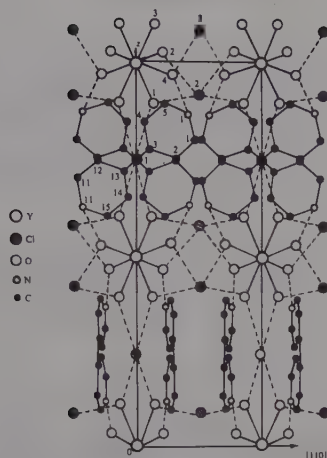
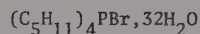
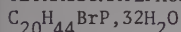


Fig. 1.  $[Y(H_2O)_8]Cl_3 \cdot 2C_{10}H_8N_2$ : a view of the unit-cell contents along  $[110]$ .

1. Preceding Report.

#### TETRAISOAMYLPHOSPHONIUM BROMIDE POLYHYDRATE



S.F. SOLODOVNIKOV, T.M. POLYANSKAYA, V.I. ALEKSEEV, L.S. ALADKO, Ya.A. DYADIN and V.V. BAKAKIN, 1982. *Kristallografiya*, 27, 247-254 [*Sov. Phys. Crystallogr.*, 27, 151-155].

Tetragonal,  $P\bar{4}$ ,  $a = 15.442$ ,  $c = 12.022$  Å,  $D_m = 1.069$ ,  $Z = 2$ . Mo radiation,  $R = 0.08$  for 1517 reflexions (at  $-120^\circ C$ ).

In the structure the water molecules and bromide ions form a hydrogen-bonded "host" framework, with tetraisoamylphosphonium "guest" cations in its large cavities (Fig. 1). The framework consists of empty decahedra  $D(4^25^8)$ , joined by their quadrilateral faces into infinite columns, and pentadecahedra  $Pd(5^{12}6^3)$ , joined into tetrahedral groups  $4Pd$  with P atoms at their centres, together with isoamyl chains in the Pd sections.

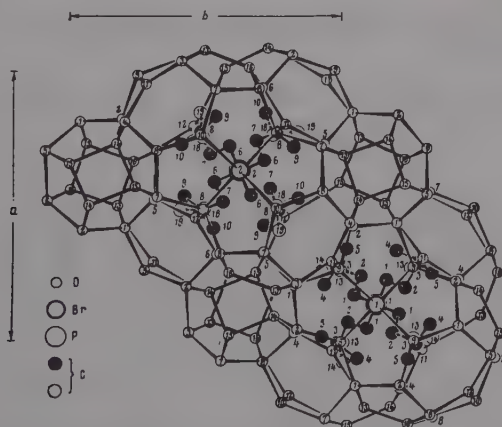


Fig. 1. Fragment of structure of  $C_{20}H_{44}BrP \cdot 32H_2O$  illustrating mutual linkage of polyhedra and relative positions of cations, projected on  $(001)$  plane.

## DEOXYCHOLIC ACID (+)-CAMPHOR



J.G. JONES, S. SCHWARZBAUM, L. LESSINGER and B.W. LOW, 1982. *Acta Cryst.*, **B38**, 1207-1215.

Orthorhombic,  $P2_12_12$ ,  $a = 27.353$ ,  $b = 13.814$ ,  $c = 7.233$  Å,  $D_m = 1.137$ ,  $Z = 4$ . Cu radiation,  $R = 0.0696$  for 2933 reflexions.

In the crystal structure (Fig. 1) the deoxycholic acid molecules form hydrogen-bonded bilayers stacked in an array which leaves hydrophobic channels between them. In these channels are columns of camphor molecules distributed between two possible orientations related by the twofold axis. The structure is compared with those of other, similar deoxycholic acid complexes.

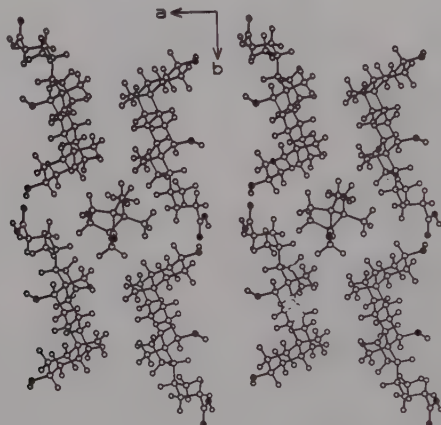
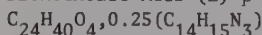


Fig. 1. A stereoscopic view of the deoxycholic acid (+)-camphor structure

## DEOXYCHOLIC ACID (E)-p-DIMETHYLAMINOAZOBENZENE (4:1 INCLUSION COMPOUND)



V.M. COIRO, E. GIGLIO, F. MAZZA, N.V. PAVEL and G. POCHETTI, 1982. *Acta Cryst.*, **B38**, 2615-2620.

Orthorhombic,  $P2_12_12_1$ ,  $a = 25.676$ ,  $b = 13.731$ ,  $c = 7.160$  Å,  $D_m = 1.17$ ,  $Z = 4$ . Mo radiation,  $R = 0.093$  for 1810 reflexions.

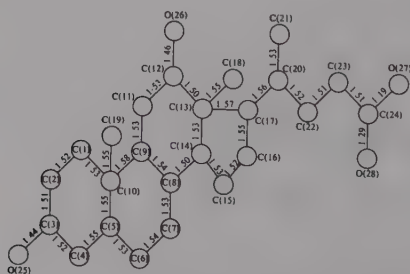


Fig. 1.  $\text{C}_{24}\text{H}_{40}\text{O}_4 \cdot \frac{1}{4}\text{C}_{14}\text{H}_{15}\text{N}_3$ : bond lengths in deoxycholic acid and a view of the crystal packing along  $c$ ; filled and open circles are methyl groups and oxygen atoms respectively and broken lines indicate hydrogen bonds.



The crystal packing is characterised by an assembly of pleated antiparallel bilayers which give rise to canals filled by p-dimethylaminoazobenzene molecules (Fig. 1). The guest molecules, located by van der Waals energy calculations, run along c and have their long axes approximately parallel to c. The C(5), C(6), C(19), C(20), C(21) and C(22) atoms, together with their H atoms, are engaged in strong interactions with the atoms of p-dimethylaminoazobenzene.

BIS(ISOTHIOCYANATO)TETRAKIS(4-METHYLPYRIDINE)NICKEL(II) NAPHTHALENE (1:2)  
 $C_{26}H_{28}N_6NiS_2 \cdot 2C_{10}H_8$   $[Ni(C_6H_7N)_4(NCS)_2]_2 \cdot 2C_{10}H_8$

J. LIPKOWSKI, 1982. *Acat Cryst.*, B38, 1745-1749.

Monoclinic, C2/c,  $a = 16.266$ ,  $b = 16.456$ ,  $c = 31.929$  Å,  $\beta = 89.26^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.065$  for 4402 reflexions.

This clathrate complex has a layer-type packing of asymmetric host molecules (Fig. 1) forming three different types of cavity. Two of these have twofold axial symmetry and are occupied by well-ordered naphthalene molecules arranged with their long axes alternately parallel and perpendicular to the twofold axis. The other cavities are centrosymmetric and are occupied by pairs of naphthalene molecules with significant disordering. The Ni atoms have distorted octahedral coordination with mean Ni-N distances 2.124(3)-(4-MePy) and 2.076(4) Å (NCS). The isothiocyanate groups are trans.

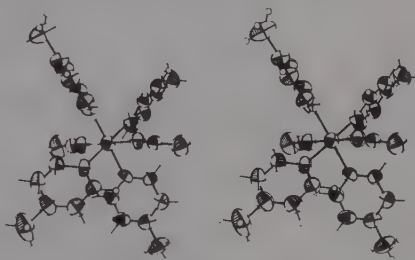


Fig. 1. Stereoview of the bis(isothiocyanato)tetrakis(4-methylpyridine)nickel(II) host molecule.

BIS(ISOTHIOCYANATO)TETRAKIS(4-METHYLPYRIDINE)NICKEL(II) 1-METHYLNAPHTHALENE (1:2)  
 $C_{26}H_{28}N_6NiS_2 \cdot 2C_{11}H_{10}$   $Ni(C_6H_7N)_4(NCS)_2 \cdot 2(CH_3C_{10}H_7)$

J. LIPKOWSKI, P. SGARABOTTO and G.D. ANDREETTI, 1982. *Acta Cryst.*, B38, 416-421.

Monoclinic,  $P2_1/c$ ,  $a = 11.528$ ,  $b = 11.890$ ,  $c = 32.852$  Å,  $\beta = 94.28^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.069$  for 5351 reflexions.

The structure of this clathrate, inclusion compound consists of discrete, propeller-shaped,  $Ni(4\text{-methylpyridine})_4(NCS)_2$  host molecules (Fig. 1) arranged in layers perpendicular to C. The (NCS) groups are nearly perpendicular to these layers and subdivide the interlayer space into a cage-like structure. The host molecules are asymmetric as shown by Ni-N(5) 2.151(4), Ni-N(6) 2.119(4) Å, N(1)-Ni-N(5) 87.9(2), Ni-N(1)-C(1) 167.6(4), Ni-N(2)-C(2) 174.5(4)° and the torsion angles C(1)-N(1)-Ni-N(3) 92(1) and C(2)-N(2)-Ni-N(3) -131(1)°. The four N atoms of the rings and Ni are coplanar; the pyridine ring planes make angles with this plane in the range 45.9-56.7°. There are two symmetrically independent guest molecules per host molecule.



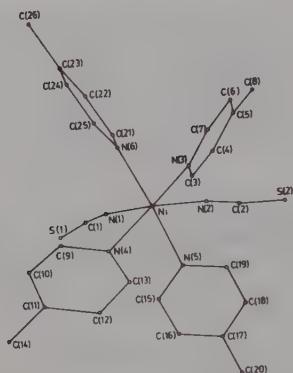


Fig. 1. The  $\text{Ni}(\text{C}_6\text{H}_7\text{N})_4(\text{NCS})_2$  molecule.

# TRI-*o*-THYMOTIDE 2-BROMOBUTANE (2:1) CLATHRATE

$\text{C}_{33}\text{H}_{36}\text{O}_6 \cdot 0.5(\text{C}_4\text{H}_9\text{Br})$

J. ALLEMAND and R. GERDIL, 1982. *Acta Cryst.*, **B38**, 1473-1476.

Trigonal,  $P\bar{3}_121$ ,  $a = 13.620$ ,  $c = 30.075$ ,  $D_m = 1.192$ ,  $Z = 6$ . Mo radiation,  $R = 0.081$  for 2286 reflexions (at 125 K).

The 2-bromobutane molecule is in a position of twofold disorder within the cage consisting of (M)-tri-*o*-thymotide molecules (Fig. 1).

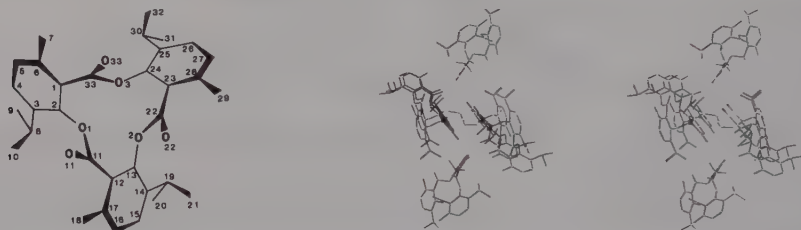


Fig. 1. The tri-*o*-thymotide molecule and a stereoview of part of the crystal structure.

# ENNIATIN B HYDRATE

$\text{C}_{33}\text{H}_{57}\text{N}_3\text{O}_9 \cdot 1.5(\text{H}_2\text{O})$

G.N. TISHCHENKO, A.I. KARAUOV and Z. KARIMOV, 1982. *Cryst. Struct. Comm.*, **11**, 451-456.

Rhombohedral,  $R\bar{3}$ ,  $a = 9.921 \text{ \AA}$ ,  $\alpha = 98.22^\circ$ . Cu radiation,  $R = 0.06$  for 877 reflexions.

The molecule (Fig. 1) has crystallographic threefold symmetry and a pseudo centre of symmetry; the two water molecules (one with 0.5 occupancy) lie on the threefold axis. There is an up-down alternation of the carbonyls with respect to the mean plane of the ring, which has the form of a strongly flattened thick-walled cup with the inner cavity of  $2.7 \text{ \AA}$  diameter. The lateral surface of the cavity is covered by the atoms of the main chain, while its bottom is composed of methyl groups of N-methylvalyl residues. The carbonyl O atoms of the hydroxy acid residues form the polar edge of the cavity which is screened by isopropyl groups. The external lipophilic surface of the molecular cup is composed of isopropyl side chains and

N-methyl groups and is incrustated by carbonyl O atoms of N-methylvalyl residues and by slightly buried N atoms of the same residues.

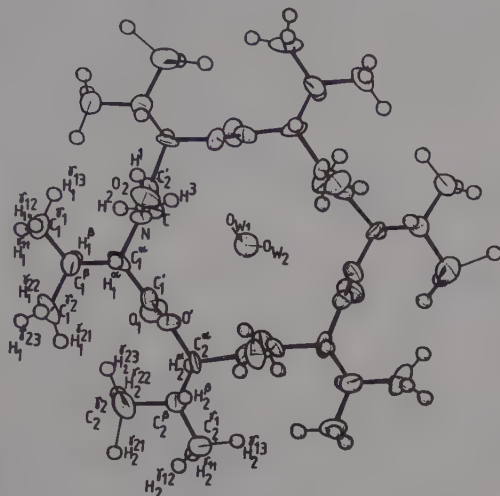


Fig. 1. The enniatin B hydrate structure.

25,26,27,28,29-PENTAHYDROXYCALIX[5]ARENE BIS(ACETONE)  
 $C_{35}H_{30}O_5 \cdot 2(C_3H_6O)$

M. CORUZZI, G.D. ANDREETTI, V. BOCCHI, A. POCHINI and R. UNGARO, 1982. J. Chem. Soc. Perkin II, 1133-1138.

Orthorhombic,  $Pna2_1$ ,  $a = 22.916$ ,  $b = 10.341$ ,  $c = 14.723$  Å,  $Z = 4$ . Mo radiation,  $R = 0.053$  for 1443 reflexions.

The chalice-like conformation of the molecule (Fig. 1) has  $C_s$  symmetry and is determined by intramolecular hydrogen bonding. The separation of adjacent phenolic oxygens ranges from 2.828(10) to 2.856(10) Å. The angle at the methylene carbons ranges from 113.2(7) to 118.1(13)° and the inclination of the phenolic units to the

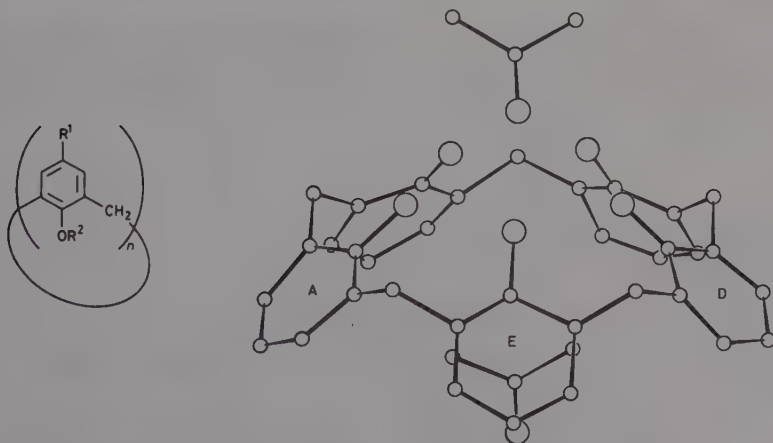
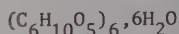
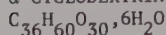


Fig. 1. Molecular skeleton ( $n = 5$ ,  $R^1 = R^2 = H$ ) and structure of  $C_{35}H_{30}O_5 \cdot 2(C_3H_6O)$ .

mean plane of the methylenes ranges from 126.5 to 139.4°. One of the acetone molecules interacts strongly with the OH groups, and the =O...O- distances which range from 2.907 to 3.139 Å, are significantly shorter than the sum of the van der Waals radii. The dihedral angle between this acetone and the mean plane of the phenolic oxygens is 126.1°. The second acetone lies within the chalice, the contacts between the methyls and rings B and C being compatible with the sum of the van der Waals radii.

$\alpha$ -CYCLODEXTRIN HEXAHYDRATE (FORM II)



K. LINDNER and W. SAENGER, 1982. *Acta Cryst.*, **B38**, 203-210.

Orthorhombic,  $P2_12_12_1$ ,  $a = 13.70$ ,  $b = 29.35$ ,  $c = 11.92$  Å,  $Z = 4$ . Cu radiation,  $R = 0.038$  for 4860 reflexions.

Form (I) of the title compound has previously been studied (1). In both crystal forms the conformation of the  $\alpha$ -cyclodextrin molecule is distorted in the same way, with one glucose moiety rotated out of alignment with the other glucose units. In form (II) (Fig. 1) one water molecule and one hydroxyl group of a symmetry-related  $\alpha$ -cyclodextrin molecule are included in the cavity while five water molecules are located outside. It is concluded that the distortion of the  $\alpha$ -cyclodextrin molecule is intrinsically associated with the 'empty' water complex and that upon complexation with guest molecules  $\alpha$ -cyclodextrin changes its conformation to a 'round, undistorted' molecule with a ring of hydrogen bonds fully established, a model for the 'induced-fit'-type complex formation.

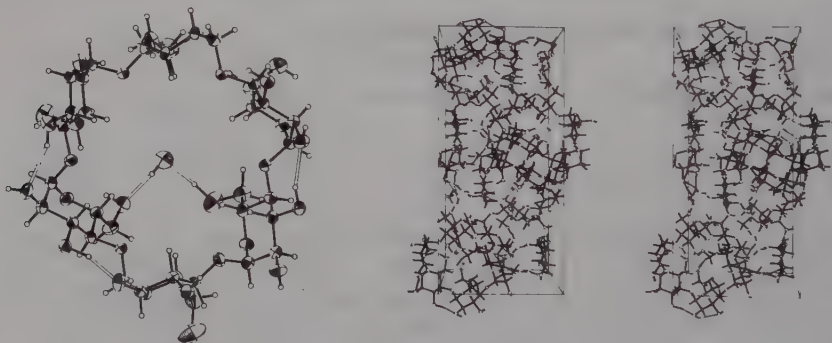
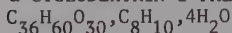


Fig. 1.  $(\text{C}_6\text{H}_{10}\text{O}_5)_6, 6\text{H}_2\text{O}$  (Form II): a perspective view of the  $\alpha$ -cyclodextrin molecule showing the water molecule enclosed in the cavity and a stereoscopic view of the unit cell, viewed down the  $c$  axis, in which hydrogen bonds are indicated by open lines, interatomic bonds by heavy lines.

1. *Structure Reports*, **46B**, 448.

$\alpha$ -CYCLODEXTRIN 1-PHENYLETHANOL TETRAHYDRATE



K. HARATA, 1982. *Bull. Chem. Soc. Jpn.*, **55**, 1367-1371.

Monoclinic,  $P2_1$ ,  $a = 8.176$ ,  $b = 23.930$ ,  $c = 13.853$  Å,  $\beta = 106.69^\circ$ ,  $D_m = 1.577$ ,  $Z = 2$ . Cu radiation,  $R = 0.040$  for 3565 reflexions.

$\alpha$ -Cyclodextrin molecules (Fig. 1) are stacked along the *a* axis in the head-to-tail fashion to form a channel-type structure, and are tilted by  $17.0^\circ$  against the channel axis. Owing to the lateral shift of  $\alpha$ -cyclodextrin ring parallel to the molecular plane, neighbouring  $\alpha$ -cyclodextrin molecules along the channel are linked by only one weak hydrogen bond. The 1-phenylethanol molecule is sandwiched between stacked  $\alpha$ -cyclodextrin molecules. S-(-) and R-(+) isomers of 1-phenylethanol are each located at the same position with the statistical disorder of the hydroxyl group with the occupancy of 0.5. Columns formed by the stack of  $\alpha$ -cyclodextrin are closely packed, being linked with one another by many hydrogen bonds. Water molecules are located in two kinds of intermolecular spaces between  $\alpha$ -cyclodextrin columns.

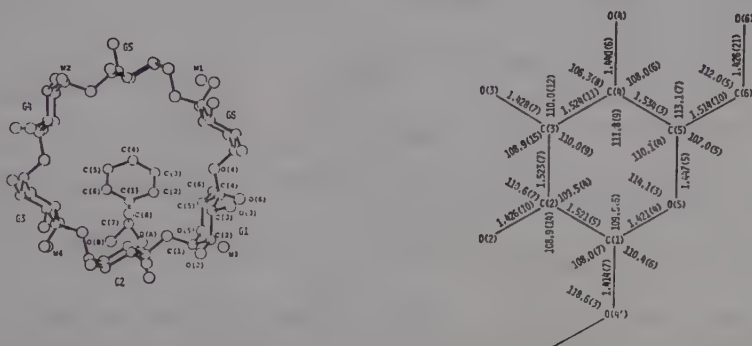
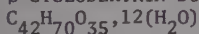


Fig. 1. A view of the  $\alpha$ -cyclodextrin complex and average bond distances for the six glucose residues.

#### $\beta$ -CYCLODEXTRIN DODECAHYDRATE



K. LINDNER and W. SAENGER, 1982. *Carbohydr. Res.*, **99**, 103-115.

Monoclinic,  $P2_1$ ,  $a = 21.29$ ,  $b = 10.33$ ,  $c = 15.10$  Å,  $\beta = 112.3$ ,  $D_m = 1.43$ ,  $Z = 2$ . Cu radiation,  $R = 0.073$  for 5189 reflexions.

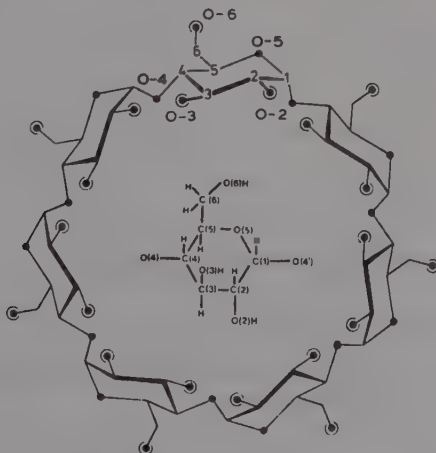


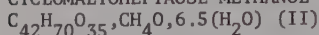
Fig. 1. The chemical structure of  $\beta$ -cyclodextrin.

The crystal structure belongs to the cage type. The  $\beta$ -CD macrocycle (Fig. 1) exists in an open, circular conformation stabilised by intramolecular hydrogen bonds between HO2 and HO3 of adjacent glucosyl residues; four of the seven HO6 groups are in the favoured (-)gauche orientation with respect to O5, two are in the (+)gauche orientation, and one is disordered over these two orientations. The 6.5 water molecules within the cavity are distributed over 8 sites and display extensive thermal motion which is probably correlated with statistical disorder.

CYCLOMALTOHEPTAOSE HYDROGEN IODIDE HYDRATE



CYCLOMALTOHEPTAOSE METHANOL HYDRATE



K. LINDNER and W. SAENGER, 1982. Carbohydr. Res., 107, 7-16.

I. Monoclinic,  $P2_1$ ,  $a = 21.25$ ,  $b = 10.28$ ,  $c = 15.30 \text{ \AA}$ ,  $\beta = 113.25^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.065$  for 5036 reflexions.

II. Monoclinic,  $P2_1$ ,  $a = 21.03$ ,  $b = 10.11$ ,  $c = 15.33 \text{ \AA}$ ,  $\beta = 111.02^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.07$  for 4643 reflexions.

The compounds are isomorphous, belong to the cage type, and have the  $\beta$ -CD molecules in nearly identical round shapes. In the HI complex (Fig. 1), one  $I^-$  is located inside, and one outside, the  $\beta$ -CD cavity; in the MeOH complex, the MeOH is within the cavity. The cavity is closed at the O2,O3 side by adjacent  $\beta$ -CD molecules, and at the O6 side by water molecules hydrogen bonded to the guest and to surrounding  $\beta$ -CD molecules. Interstices between  $\beta$ -CD molecules are filled by water of hydration molecules in distorted coordination.

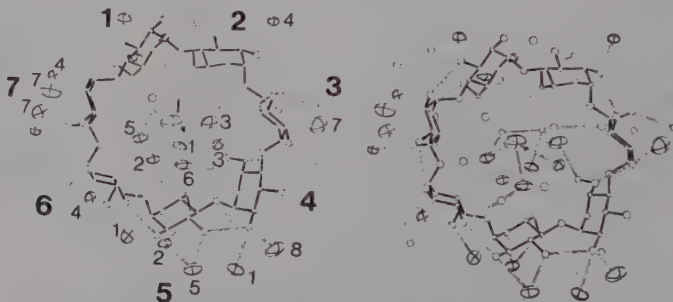


Fig. 1. A stereoview of the  $\beta$ -CD, 2HI, 8H<sub>2</sub>O complex.

$\beta$ -CYCLODEXTRIN 1,4-DIAZABICYCLO[2.2.2]OCTANE TRIDECAHYDRATE



K. HARATA, 1982. Bull. Chem. Soc. Jpn., 55, 2315-2320.

Monoclinic,  $P2_1$ ,  $a = 15.395$ ,  $b = 16.598$ ,  $c = 15.441 \text{ \AA}$ ,  $\beta = 117.35^\circ$ ,  $D_m = 1.40$ ,  $Z = 2$ . Cu radiation,  $R = 0.048$  for 5649 reflexions.

The  $\beta$ -cyclodextrin molecule (Fig. 1) is in the shape of a distorted and truncated heptagonal pyramid. Two of seven primary hydroxyl groups are in a gauche-trans conformation, the others being in a gauche-gauche conformation. Secondary hydroxyl groups form intramolecular hydrogen bonds between adjacent glucose residues. The guest 1,4-diazabicyclo[2.2.2]octane is almost fully included in the cavity of  $\beta$ -cyclodextrin. Its methylene groups are in van der Waals contact with the methine groups and the glycosidic oxygen atoms at the interior surface of  $\beta$ -cyclodextrin, while two nitrogen atoms form hydrogen bonds with a water molecule and a secondary hydroxyl group of the neighbouring  $\beta$ -cyclodextrin.  $\beta$ -Cyclodextrin molecules are arranged nearly parallel to the crystallographic ac plane to form a corrugated sheet of molecular layer. All of the thirteen water molecules fill vacant spaces between the molecular layers, forming a hydrogen-bond network in the crystal.

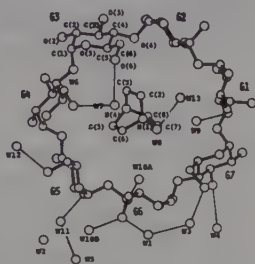


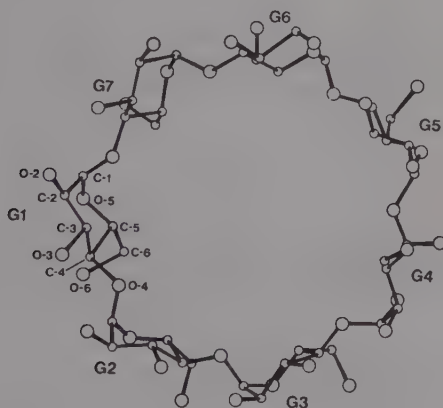
Fig. 1. A view of the  $\beta$ -cyclodextrin complex.

# CYCLOMALTOHEPTAOSE BENZOCAINE HYDRATE

$C_{42}H_{70}O_{35} \cdot C_9H_{11}NO_2 \cdot 7.5(H_2O)$

J.A. HAMILTON and M.N. SABESAN, 1982. Carbohydr. Res., **102**, 31-46.

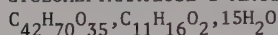
Monoclinic, C2,  $a = 18.746$ ,  $b = 24.528$ ,  $c = 15.658$  Å,  $\beta = 110.21^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.16$  for 3058 reflexions.





The  $\beta$ -CD molecule (Fig. 1) exists as a dimer in the crystal by means of extensive hydrogen bonding across the secondary-hydroxyl ends of two cyclomaltoheptaose molecules. A continuous channel throughout the crystal is achieved by stacking of these dimer units. The two halves of the  $\beta$ -CD dimer are related by the crystallographic, twofold axis, but the benzocaine guests in each half do not follow this symmetry, and are statistically disordered. The ester groups are at the secondary-hydroxyl end of the  $\beta$ -CD molecule and the guests are disordered (to avoid overcrowding around the crystallographic, twofold axis). A lone, water molecule is trapped in the cavity at the secondary hydroxyl interface, and forms a link between the ester groups of the two guest molecules by hydrogen bonding to the carbonyl oxygen atom of both. The p-amino group of each guest in the dimer pair has a different, and independent, hydrogen-bonding scheme. One amino group hydrogen-bonds directly to a primary hydroxyl group of the  $\beta$ -CD (in the gauche-trans position), whereas the other is hydrogen-bonded via a water molecule in the cavity at the primary-hydroxyl end to a different primary hydroxyl group.

# CYCLOHEPTAAMYLOSE 1-ADAMANTANECARBOXYLIC ACID HYDRATE



J.A. HAMILTON and M.N. SABESAN, 1982. *Acta Cryst.*, B38, 3063-3069.

Triclinic, P1,  $a = 17.747$ ,  $b = 15.255$ ,  $c = 15.491$  Å,  $\alpha = 102.54$ ,  $\beta = 113.54$ ,  $\gamma = 98.87^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.11$  for 6433 reflexions.

Two independent molecules of each of cycloheptaamylose and 1-adamantanecarboxylic acid per unit cell form a dimer (Fig. 1) of composition  $2\text{C}_{11}\text{H}_{16}\text{O}_2, 2\text{C}_{42}\text{H}_{70}\text{O}_{35}, 30\text{H}_2\text{O}$ . Two cycloheptaamylose molecules are in a head-to-head dimer produced by tight hydrogen bonding involving the secondary hydroxyl groups on both molecules. The two 1-adamantanecarboxylic acid guests have different orientations in the cycloheptaamylose cavities and different depths of penetration.

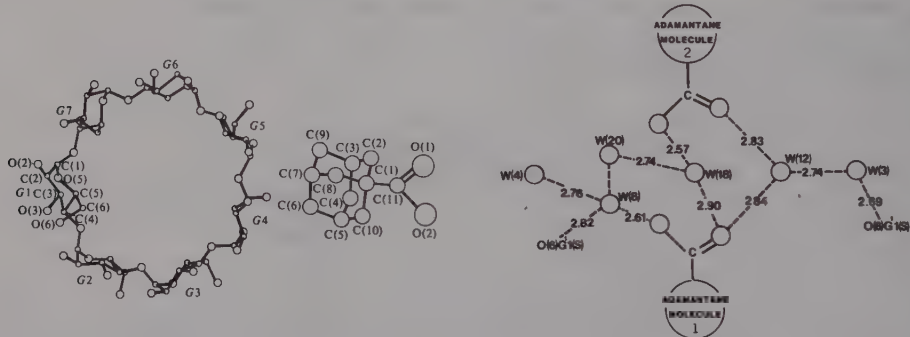
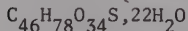


Fig. 1. Views of an adamantyl and cycloheptaamylose residue and a schematic diagram showing how molecules are linked by hydrogen bonds.

# 6-DIOXY-6-(t-BUTYLTHIO)- $\beta$ -CYCLODEXTRIN HYDRATE



K. HIROTSU, T. HIGUCHI, K. FUJITA, T. UEDA, A. SHINODA, T. IMOTO and I. TABUSHI, 1982. *J. Org. Chem.*, 47, 1143-1144.

Orthorhombic,  $P2_12_12_1$ ,  $a = 32.107$ ,  $b = 15.517$ ,  $c = 15.097$  Å,  $Z = 4$ . Mo radiation,  $R = 0.130$  for 3497 reflexions (at  $-100^\circ\text{C}$ ).

The molecules of this complex (Fig. 1) are located around a  $2_1$  axis to give a polymeric structure where the tert-butylthio group (guest) is intermolecularly included in the hydrophobic cavity of the cyclodextrin moiety (host), directly confirming that the monosubstituted cyclodextrin includes the guest in its cavity. The cyclodextrin moiety has an approximate 7-fold axis and a round shape, demonstrating that the monosubstitution does not particularly alter the structure of the cyclodextrin macrocycle compared.

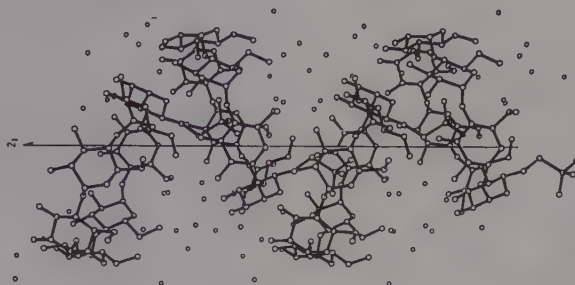


Fig. 1. A drawing of the  $C_{46}H_{78}O_{34}S, 22H_2O$  inclusion complex. The small circles are water molecules.

HEXAKIS(2,3,6-TRI-O-METHYL)- $\alpha$ -CYCLODEXTRIN p-NITROPHENOL MONOHYDRATE  
 $C_{54}H_{96}O_{30}, C_6H_5NO_3, H_2O$

K. HARATA, K. UEKAMA, M. OTAGIRI and F. HIRAYAMA, 1982. Bull. Chem. Soc. Jpn., 55, 3904-3910.

Monoclinic,  $P2_1$ ,  $a = 11.307$ ,  $b = 14.578$ ,  $c = 22.118$  Å,  $\beta = 93.62^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.048$  for 4976 reflexions.

The methyl- $\alpha$ -CDx molecule (Fig. 1) has the shape of a truncated hexagonal cone, into which the p-nitrophenol molecule is inserted. The phenolic hydroxyl group is located at the centre of the methyl- $\alpha$ -CDx cavity, while the nitro group protrudes outside from the base-side of the host cone and is in contact with the adjacent methyl- $\alpha$ -CDx molecule. The water molecule, which is also located within the cavity, links the methyl- $\alpha$ -CDx and p-nitrophenol molecules by hydrogen bonds. Methyl- $\alpha$ -CDx molecules are stacked along the crystallographic a axis in a head-to-tail mode to form a channel-type structure.

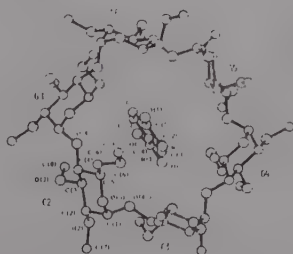


Fig. 1. A view of methyl- $\alpha$ -CDx:p-nitrophenol monohydrate complex.

HEXAKIS(2,3,6-TRI-O-METHYL)- $\alpha$ -CYCLODEXTRIN p-iodoaniline MONOHYDRATE  
 $C_{54}H_{96}O_{30}, C_6H_4IN, H_2O$

K. HARATA, K. UEKAMA, M. OTAGIRI and F. HIRAYAMA, 1982. Bull. Chem. Soc. Jpn., 55, 407-410.

Monoclinic,  $P2_1$ ,  $a = 11.440$ ,  $b = 23.674$ ,  $c = 13.531$  Å,  $\beta = 91.90^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.11$  for 2186 reflexions.

The  $\alpha$ -cyclodextrin moieties form a truncated elliptical cone and these cones are stacked along the  $a$ -axis to form a channel-type structure. The guest  $p$ -iodoaniline and water molecules are located in the channel. The iodine is at the centre of the cavity while the amino group (outside the cavity) forms a hydrogen bond with the water molecule which is in turn hydrogen bonded to the adjacent host molecule along the channel.

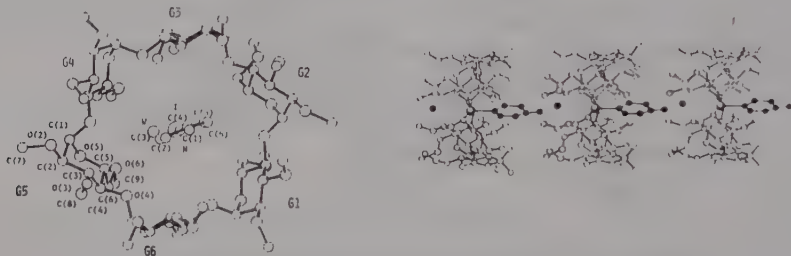


Fig. 1. Views of the  $C_{54}H_{96}O_{30}$ ,  $C_6H_6IN$ ,  $H_2O$  host-guest structure.

HEXAKIS(2,3,6-TRI-O-METHYL)- $\alpha$ -CYCLODEXTRIN BENZALDEHYDE  
 $C_{54}H_{96}O_{30}$ ,  $C_7H_6O$

K. HARATA, K. UEKAMA, M. OTAGIRI, F. HIRAYAMA and Y. SUGIYAMA, 1982. Bull. Chem. Soc. Jpn., 55, 3386-3389.

Monoclinic,  $P2_1$ ,  $a = 11.604$ ,  $b = 23.832$ ,  $c = 13.539$  Å,  $\beta = 106.11^\circ$ ,  $D_m = 1.22$ ,  $Z = 2$ . Cu radiation,  $R = 0.10$  for 3597 reflexions.

The methyl- $\alpha$ -CDx molecule (Fig. 1) is in the shape of a distorted and truncated hexagonal cone. The guest benzaldehyde molecule is fully included within the host cavity. The carbonyl group is located at the centre of the cavity, while the phenyl group is located at the base-side of the host cone. Methyl- $\alpha$ -CDx molecules, of which pseudo hexagonal axis is inclined by an angle of  $17.0^\circ$  against the crystallographic  $a$  axis, are stacked along the  $a$  axis to form a head-to-tail channel-type structure.

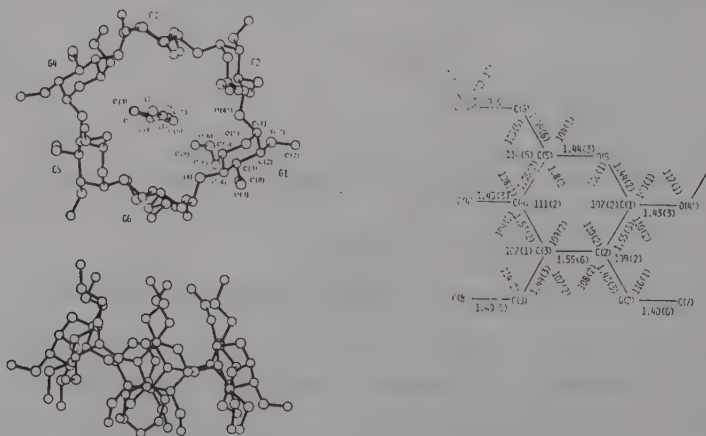


Fig. 1. Views of the  $\alpha$ -CDx-benzaldehyde complex and average dimensions for the glucose residue.

POTASSIUM METHYLTRIFLUOROBORATE  
 $\text{CH}_3\text{BF}_3\text{K}$



D.J. BRAUER, H. BÜRGER and G. PAWELKE, 1982. *J. Organometal. Chem.*, **238**, 267-279.

Orthorhombic,  $\text{Pbca}$ ,  $a = 17.172$ ,  $b = 7.1486$ ,  $c = 7.2289 \text{ \AA}$ ,  $Z = 8$ . Mo radiation,  $R = 0.026$  for 933 reflexions.

The cations form their closest contacts with F atoms in five anions, the midpoints of which roughly describe a square pyramid (Fig. 1). Seven K-F contacts are found at 2.678 to 2.965 Å. The symmetry of the  $\text{CH}_3\text{BF}_3^-$  anion is approximately  $\text{C}_{3v}$  with the  $\text{CH}_3$  group staggered with respect to the  $\text{BF}_3$  fragment. The B-C bond length (1.575 Å) is very short in contrast to the B-F distances (1.434, 1.418 and 1.420 Å) which are considered long for fluoroborates.

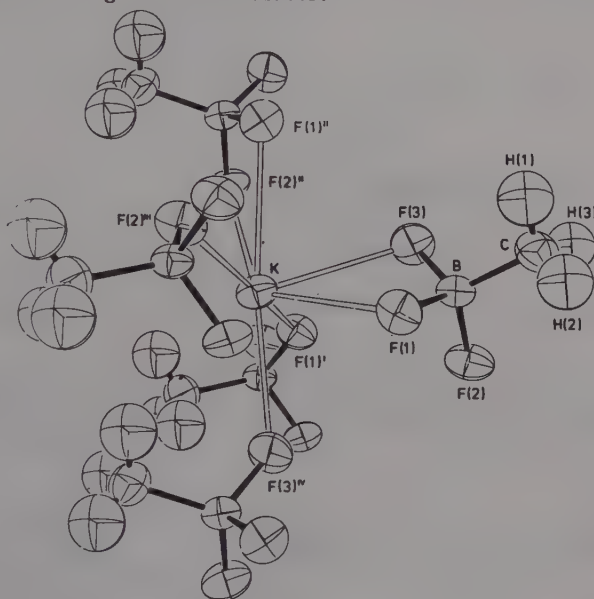
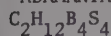


Fig. 1. View of the cation and five closest anions in  $\text{K}^+ \cdot [\text{CH}_3\text{BF}_3]^-$ .

1,3,5,7-TETRATHIA-2,4,6,8-TETRABORATRICYCLO(3.3.1.1<sup>3,7</sup>)DECANE (BORON SULPHUR  
 ADAMANTANE)



H. BINDER, W. DIAMANTIKOS, K. DERMENTZIS and H.-D. HAUSEN, 1982. *Z. Naturforsch.*, **37b**, 1548-1552.

Monoclinic,  $\text{P2}_1/\text{c}$ ,  $a = 7.307$ ,  $b = 9.115$ ,  $c = 14.626 \text{ \AA}$ ,  $\beta = 90.84^\circ$ ,  $D_m = 1.40$ ,  $Z = 4$ . Mo radiation,  $R = 0.027$  for 2240 reflexions (at  $-100^\circ\text{C}$ ).

The structure was confirmed as that shown in Fig. 1. Mean values for bond lengths are: S-C 1.821(4), S-B 1.926(5) Å.

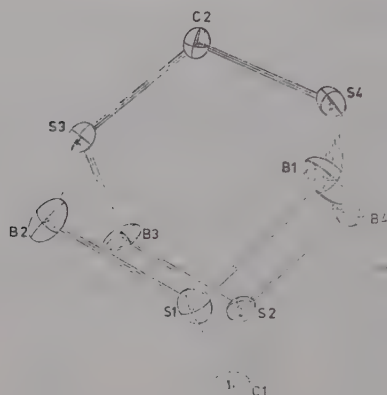
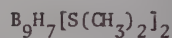
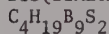


Fig. 1. The structure of  $(\text{CH}_2(\text{SBH}_2)_2)_2$ .

BIS(DIMETHYL SULPHIDE)-closo-NONABORANE(7)



D.D. BRAY, R.M. KABBANI and E.H. WONG, 1982. *Acta Cryst.*, **B38**, 957-959.

Orthorhombic,  $\text{P}2_12_12_1$ ,  $a = 7.879$ ,  $b = 9.014$ ,  $c = 19.436 \text{ \AA}$ ,  $Z = 4$ . Cu radiation,  $R = 0.079$  for 1538 reflexions.

In the molecule (Fig. 1) the nine-boron cage approximates to the tricapped trigonal prismatic geometry common to  $\text{B}_9$  boranes. One dimethyl sulphide ligand is bonded to a capping boron and the other to a trigonal prismatic boron atom; B-S distances are 1.848 and 1.891  $\text{\AA}$  respectively.

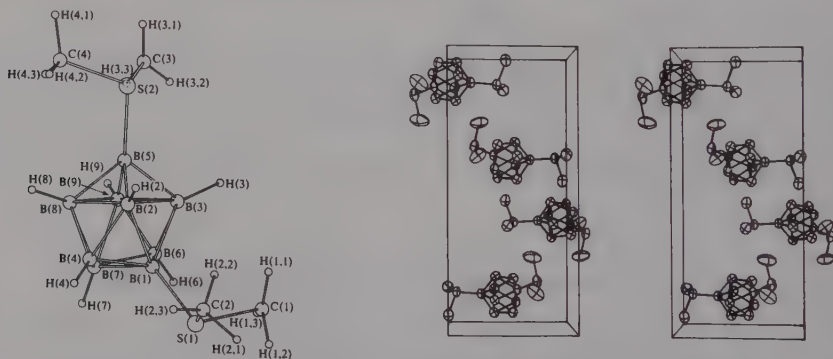
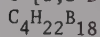


Fig. 1.  $\text{B}_9\text{H}_7[\text{S}(\text{CH}_3)_2]_2$ : a perspective view of the molecule (left) and a stereoscopic drawing of the contents of one unit cell (right).

8-[1,2-DICARBA-closo-DODECABORAN(12)-3-YL]-5,6-DICARBA-nido-DECABORANE(12)



V. SUBRTOVA, A. LINEK and J. HASEK, 1982. *Acta Cryst.*, **B38**, 3147-3149.

Monoclinic,  $\text{C}2/c$ ,  $a = 21.099$ ,  $b = 7.081$ ,  $c = 22.065 \text{ \AA}$ ,  $\beta = 94.27^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.054$  for 2108 reflexions.

Fig. 1 shows a view of the molecule. The X-ray study confirms the suggestion that the structure consists of two different carbaborane frameworks bonded together by a B-B bond of 1.681(2) Å between B(8') of the nido moiety and B(3) of the closo-1,2-dicarbaborane. The mutual orientation of the two frameworks is defined by the torsion angles C(1)-B(3)-B(8')-B(7') 39.8, C(2)-B(3)-B(8')-B(7') 105.6 and C(2)-B(3)-B(8')-B(3') 38.1°. The 5,6-dicarba-nido-decaborane framework,  $C_2H_{11}B_8$ , is substantially distorted due to the two C heteroatoms in the 5' and 6' positions. Between these C atoms a very short distance, 1.457(2) Å, was found. The C-B distances in this part of the molecule range from 1.508(2) to 1.751(2) Å, the average B-B distance being 1.793(47) Å. The bond lengths in the 1,2-dicarbaborane icosahedron,  $C_2H_{11}B_{10}$ , agree well with those in related icosahedral carbaboranes. The average B-B and B-C distances are 1.774(9) and 1.708(16) Å.

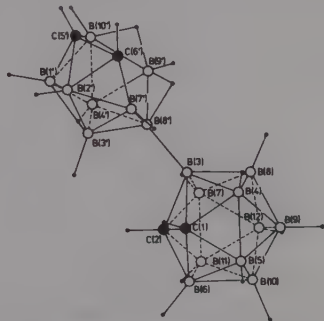
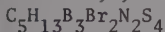


Fig. 1. The structure and numbering scheme for  $C_4H_{22}B_{18}$ .

(2,4,6-EPITRITHIO)-2,6-DIBROMO-3-(ETHYLENE-1,2-DIYL)-4,5,5-TRIMETHYL-1-THIA-3,5-DIAZA-2,4,6-TRIBORACYCLOHEXANE



H. NÖTH and R. STAUDIGL, 1982. Chem. Ber., 115, 813-817.

Monoclinic,  $P2_1/c$ ,  $a = 8.816$ ,  $b = 11.952$ ,  $c = 11.408$  Å,  $\beta = 106.29^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.080$  for 1998 reflexions.

The structure, which has approximate  $C_s$  symmetry, was confirmed as that shown in Fig. 1. Principal bond lengths are: B-S(3) 1.88(1), B(1)-S(1)B(2)-S(4) 1.92(1), B-N 1.60(2), B(3)-S(2) 2.04(1), S-S 2.036(4) Å.

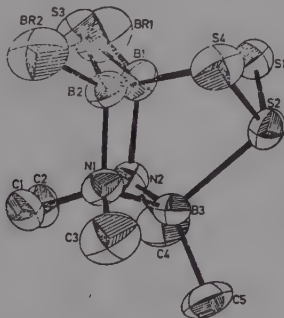


Fig. 1. Perspective view of  $C_5H_{13}B_3Br_2N_2S_4$ .



8,8'- $\mu$ -METHOXO-3,3'-commo-BI[1,2-DICARBA-3-FERRA-closo-DODECABORANE(11)]  
 $C_5H_{23}B_{18}FeO$   $OCH_3(B_9C_2H_{10})_2Fe$

V. ŠUBRTOVÁ, K. MALÝ, V. PETŘÍČEK and A. LÍNEK, 1982. Acta Cryst., B38, 2028-2031.

Orthorhombic,  $P2_12_12_1$ ,  $a = 12.128$ ,  $b = 21.521$ ,  $c = 6.991$  Å,  $Z = 4$ . Mo radiation,  $R = 0.056$  for 2167 reflexions.

In the molecule (Fig. 1) the planes C(1),C(2),B(4),B(7),B(8) and C(1'),C(2'),-B(4'),B(7'),B(8') are inclined at  $31.2(5)^\circ$  and the perpendicular Fe-plane distances are both 1.531(6) Å. The B(5),B(6),B(11),B(12),B(9) and B(5'),B(6'),B(11'),B(12'),-B(9') planes are inclined at  $28.9^\circ$ . The Fe-C distances are in the range 2.159(5)-2.176(6) Å while those of Fe-B(4),B(7),B(4'),B(7') are in the range 2.117(5)-2.133(6) Å. Mean bond lengths within the "cages" are, B-B 1.778(9), B-C 1.703(8), C-C 1.625(8), B-H 1.03(8) Å. The O-B(8') and O-B(8) distances are 1.536(6) and 1.498(7) Å respectively.

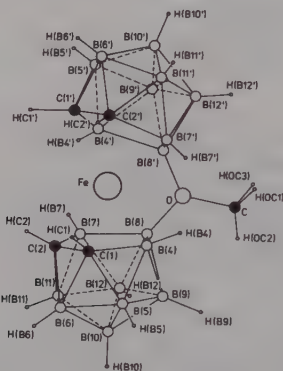


Fig. 1. The  $OCH_3(B_9C_2H_{10})_2Fe$  molecule.

# TRIS(DIMETHYLAMINO)BORANE

$C_6H_{18}BN_3$

$B(N(CH_3)_2)_3$

G. SCHMID, R. BOESE and D. BLASER, 1982. Z. Naturforsch., 37b, 1230-1233.

Monoclinic,  $P2_1/n$ ,  $a = 8.293$ ,  $b = 8.025$ ,  $c = 14.043$  Å,  $\beta = 90.41^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.047$  for 1922 reflexions (at  $-116^\circ C$ ).

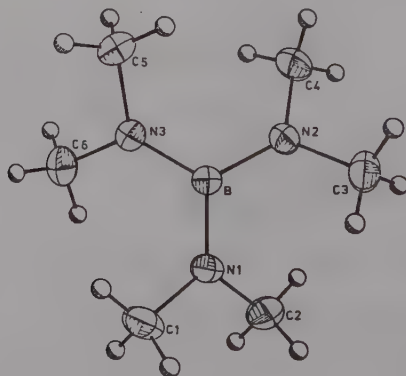


Fig. 1. The structure of  $B(N(CH_3)_2)_3$  projected onto the  $BN_3$  plane.

The  $\text{BN}_3$  moiety (Fig. 1) is planar within experimental error and the C atoms alternate above and below this plane by an average of 0.59 Å. The  $\text{NC}_2$  planes are rotated an average of  $31.3^\circ$  with respect to the  $\text{BN}_3$  plane. Principal bond lengths are: B-N 1.439(1), N-C 1.453(1) Å.

(N,N,N',N'-TETRAMETHYLETHYLENEDIAMINE) TETRABORANE

$\text{C}_6\text{H}_{24}\text{B}_4\text{N}_2$  (I)

( $\mu_2$ -1,2-BIS(DIPHENYLPHOSPHINO)ETHANE- $\text{B}^1, \text{B}^2$ ) PENTABORANE

$\text{C}_{26}\text{H}_{33}\text{B}_5\text{P}_2$  (II)

( $\mu_2$ -BIS(DIPHENYLPHOSPHINO)METHANE- $\text{B}^1, \text{B}^2$ ) PENTABORANE TETRAHYDROFURAN SOLVATE

$\text{C}_{25}\text{H}_{31}\text{B}_5\text{P}_2 \cdot 0.5(\text{C}_4\text{H}_8\text{O})$  (III)

(N,N,N',N'-TETRAMETHYLETHYLENEDIAMINE) PENTABORANE

$\text{C}_6\text{H}_{25}\text{B}_5\text{N}_2$  (IV)

N.W. ALCOCK, H.M. COLQUHOUN, G. HARAN, J.F. SAWYER and M.G.H. WALLBRIDGE, 1982. J. Chem. Soc. Dalton, 2243-2255.

I. Monoclinic, Cc,  $a = 7.654$ ,  $b = 12.937$ ,  $c = 12.536$  Å,  $\beta = 107.78^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.134$  for 576 reflexions.

II. Orthorhombic, Pbca,  $a = 14.297$ ,  $b = 16.492$ ,  $c = 22.280$  Å,  $Z = 8$ . Mo radiation,  $R = 0.047$  for 2643 reflexions.

III. Orthorhombic,  $\text{P}2_12_12_1$ ,  $a = 7.931$ ,  $b = 16.816$ ,  $c = 23.271$  Å,  $Z = 4$ . Mo radiation,  $R = 0.069$  for 1850 reflexions.

IV. Orthorhombic,  $\text{P}2_12_12_1$ ,  $a = 12.940$ ,  $b = 12.911$ ,  $c = 15.046$  Å,  $Z = 8$ . Mo radiation,  $R = 0.055$  for 1200 reflexions.

In II and III the  $\text{B}_5\text{H}_9$  moieties have become shallow pyramids (Fig. 1) with rearrangement of the H atoms to give basal ' $\text{BH}_2$ ' groups and two symmetrical H bridges. Within the  $\text{B}_5$  unit, two triangular  $\text{B}_3$  faces share a vertex (B-B 1.745-1.829, B...B 2.72(2)-2.75(2) (II), 2.723(7)-2.755(7) Å (III)). Other dimensions in the two compounds are very similar and normal. The P atoms of the dppm and dppc ligands bridge apical and basal positions of these  $\text{B}_5$  groups (P-B 1.927(9), 1.913(10) (II), 1.939(4), 1.953(4) Å (III)). The  $\text{B}_2\text{P}_2$  units are planar. The average P-C(Ph) distances are 1.828 and 1.821 Å for II and III respectively. The two independent molecules of IV each contain a  $\text{B}_5$  framework which consists of a  $\text{B}_4\text{H}_8$  unit and a single B atom. In the  $\text{B}_4\text{H}_8$  unit two  $\text{B}_3$  triangles share an edge (B-B 1.699-1.835 Å). Both N atoms of the tetramethylethylenediamine ligand donate to the single B atom to form a five-membered chelate (B-N 1.673 Å, N-B-N  $97.1^\circ$ ). One of the independent molecules is disordered in this chelate ring. The structure of I was not determined to high precision but the overall structure is clear. Again both N atoms coordinate to a single B.

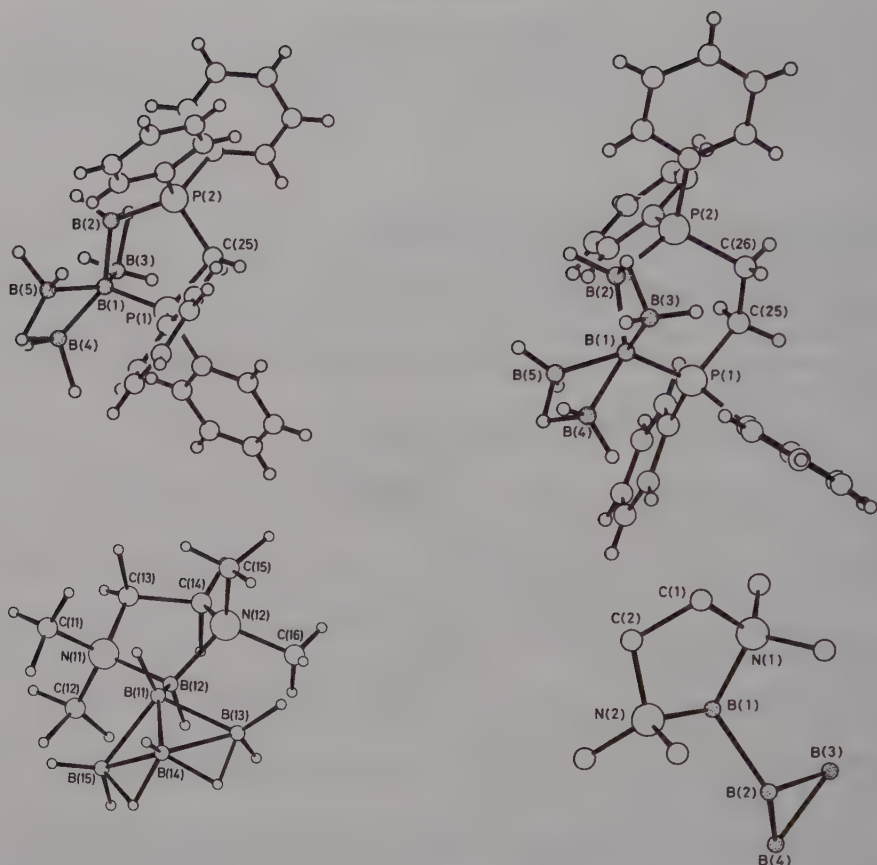


Fig. 1. The molecular structures:  $C_{26}H_{33}B_5P_2$  (top left),  $C_{25}H_{31}B_5P_2$  (top right),  $C_6H_{25}B_5N_2$  (bottom left) and  $C_6H_{24}B_4N_2$ .

BIS(ACETATO)(ACETYLACETONATO)BORON(III)  
 $C_9H_{13}BO_6$

$B(O_2CCH_3)_2(C_5H_7O_2)$

F.A. COTTON and W.H. ILSLEY, 1982. *Inorg. Chem.*, **21**, 300-302.

Monoclinic,  $P2_1/c$ ,  $a = 14.138$ ,  $b = 7.440$ ,  $c = 11.593$  Å,  $\beta = 107.60^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.035$  for 919 reflexions.

The structure consists of discrete molecules (Fig. 1) that have approximate  $C_{2v}$  symmetry. The mean B-O distances are 1.450(4) Å (acetate) and 1.471(4) Å (acetylacetonato). The six-membered ring is planar to within  $\pm 0.014$  Å.

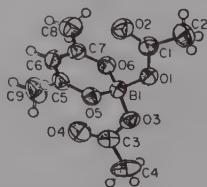


Fig. 1. The  $B(O_2CCH_3)_2(C_5H_7O_2)$  molecule.

5-p-FLUOROPHENYL-2,4,6-TRIMETHYLCYCLO-1,3-DIOXA-2,4,6-TRIBORANE  
 $C_9H_{13}B_3FNO_2$

A. MELLER, C. HABBEN, M. NOLTEMEYER and G.M. SHELDRICK, 1982. *Z. Naturforsch.*, **37b**, 1504-1506.

Orthorhombic, *Pnma*,  $a = 7.362$ ,  $b = 12.566$ ,  $c = 13.306$  Å,  $Z = 4$ . Mo radiation,  $R = 0.075$  for 554 reflexions.

The heterocyclic ring lies in a crystallographic mirror plane with the p-fluorophenyl perpendicular to it (Fig. 1). Principal bond lengths are: B-N 1.44(1), B-O 1.38(1), N(1)-C(1') 1.44(1) Å.

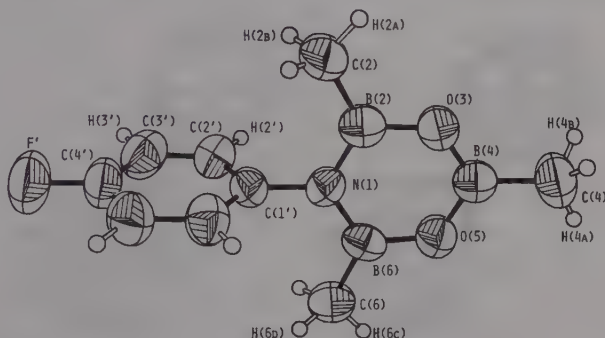


Fig. 1. The structure of  $C_9H_{13}B_3FNO_2$ .

PIPERIDINIUM TETRAMETHOXYBORATE



N.W. ALCOCK, R.M. HAGGER, W.D. HARRISON and M.G.H. WALLBRIDGE, 1982. *Acta Cryst.*, **B38**, 676-677.

Orthorhombic, *Pnma*,  $a = 13.325$ ,  $b = 9.603$ ,  $c = 10.305$  Å,  $Z = 4$ . Mo radiation,  $R = 0.051$  for 494 reflexions (at 213 K).

The structure (Fig. 1) consists of layers of discrete cations and anions, perpendicular to  $b$ , with crystallographic mirror symmetry. The ions are linked by hydrogen bonds. The B-O bond lengths are 1.475(4) (to O involved in hydrogen bonding), 1.451(3) and 1.454 Å. The O-B-O angles are in the range 101.4-114.1°.

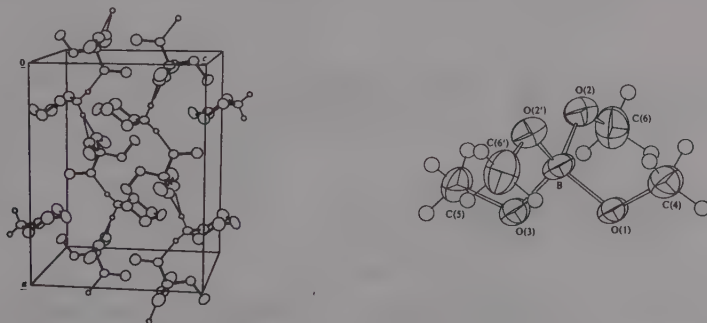


Fig. 1. The packing of  $C_9H_{24}BNO_4$  and a view of the anion.

TRIETHYLAMMONIUM BIS(1,2-DICARBA-UNDECABORANE) COBALT  
 $C_{10}H_{38}B_{18}CoN$



L. BORODINSKY, E. SINN and R.N. GRIMES, 1982. *Inorg. Chem.*, 21, 1686-1689.

Orthorhombic,  $Pna2_1$ ,  $a = 29.048$ ,  $b = 11.457$ ,  $c = 7.100$  Å,  $Z = 4$ . Mo radiation,  $R = 0.049$  for 1879 reflexions.

The anion (Fig. 1) consists of two  $C_2B_9H_{11}^{2-}$  units sandwiched around a  $Co^{3+}$  cation with the  $C_2B_3$  faces of the two ligands nearly parallel ( $3.7^\circ$  dihedral angle). The mean Co-C and Co-B distances are  $2.046(2)$  and  $2.097(2)$  Å. The two  $C_2B_9H_{11}^{2-}$  ligands are mutually rotated by  $37^\circ$ .

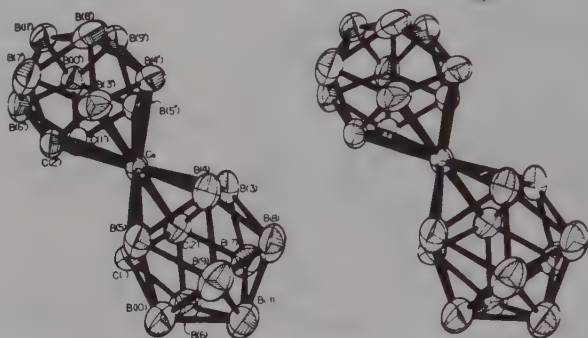
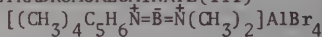


Fig. 1. Stereoview of the  $[Co(1,2-C_2B_9H_{11})_2]^{2-}$  anion.

(DIMETHYLAMIDO) (2,2,6,6-TETRAMETHYLPYPERIDINO) BORON(1+) TETRABROMOALUMINATE (III)  
 $C_{11}H_{24}AlBr_4N_2$



H. NÖTH, R. STAUDIGL and H.-U. WAGNER, 1982. *Inorg. Chem.*, 21, 706-716.

Monoclinic,  $P2_1/n$ ,  $a = 7.678$ ,  $b = 14.56$ ,  $c = 17.91$  Å,  $\beta = 97.25^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.104$  for 1851 reflexions (at  $-40^\circ C$ ).

The  $C_2NBNC_2$  skeleton of the cation (Fig. 1) is of the allene type with a nearly linear NBN atomic arrangement ( $N-B-N$   $176(3)^\circ$ ). Both B-N distances are rather short ( $1.30(4)$  and  $1.42(4)$  Å), demonstrating a fairly high bond order for the B-N bonds.

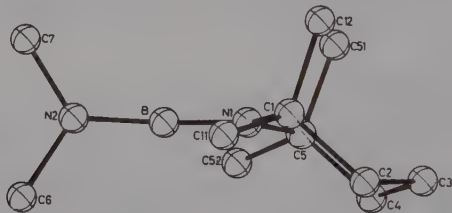
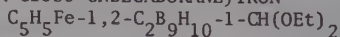


Fig. 1. The  $[(CH_3)_4C_5H_6\dot{N}=\dot{B}=\dot{N}(CH_3)_2]$  cation.

( $\eta^5$ -CYCLOPENTADIENYL) - ( $\eta^5$ -(1-DIETHOXYMETHYL)-1,2-DICARBA-closo-UNDECABORANE) IRON  
 $C_{12}H_{28}B_9FeO_2$



L.I. ZAKHARKIN, V.V. KOBAK, A.I. YANOVSKY and Yu.T. STRUCHKOV, 1982. *J. Organometal. Chem.*, 228, 119-133.

Monoclinic,  $P2_1/a$ ,  $a = 14.088$ ,  $b = 14.925$ ,  $c = 9.236$  Å,  $\beta = 104.94^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.056$  for 1599 reflexions.

The Fe atom is  $\eta^5$ -coordinated by the "open"  $C_2B_3$  face of the  $\pi$ -dicarbollyl ligand and the  $\pi$ - $C_5H_5$  ligand (Fig. 1). Its distance from these planes is 1.486 and 1.725 Å respectively. The Fe-C and Fe-B distances to the  $C_2B_3$  face are on average 2.063 and 2.100 Å respectively while the average Fe-C(cyclopentadienyl) distance is 2.08 Å. The dihedral angle between the  $C_2B_3$  and the  $C_5H_5$  planes is  $7^\circ$ . The C(2)-C(18) bond is bent towards the Fe atom and makes an angle of  $19^\circ$  with the  $C_2B_3$  plane. The shortness (1.37 Å) of the C(23)-C(24) bond may be due to disorder or intense thermal motion.

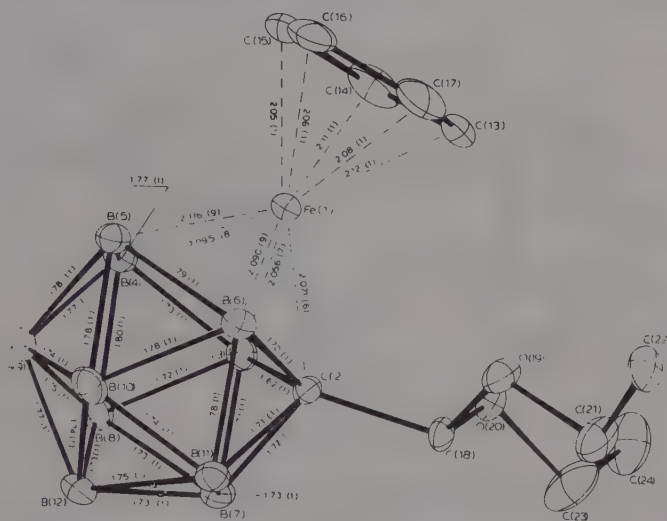


Fig. 1. Molecular structure of  $C_{12}H_{28}B_9FeO_2$  showing bond distances (Å).

# TETRAMETHYLAMMONIUM 7-PHENYL-7,8-DICARBA-nido-UNDECABORANE

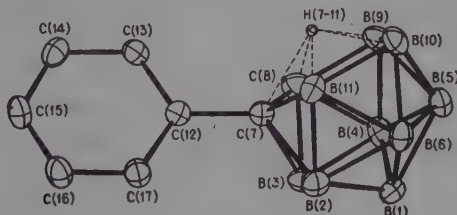
$C_{12}H_{28}B_9N$

$[(CH_3)_4N][C_6H_5C_2B_9H_{11}]$

A.I. YANOVSKII, Yu.T. STRUCHKOV, V.N. KALININ and L.I. ZAKHARKIN, 1982. Zh. Strukt. Khim., 23-2, 77-80 [J. Struct. Chem., 23, 232-235].

Monoclinic,  $P2_1$ ,  $a = 7.292$ ,  $b = 10.401$ ,  $c = 12.488$  Å,  $\beta = 101.67^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.072$  for 1277 reflexions.

The structure consists of disordered  $Me_4N^+$  cations and  $[C_6H_5C_2B_9H_{11}]^-$  anions. One of the H atoms of the anion is centered on a pentagonal open face of the carborane polyhedron (Fig. 1) and is located at a distance of 0.81(5) Å from the mean plane of this face.



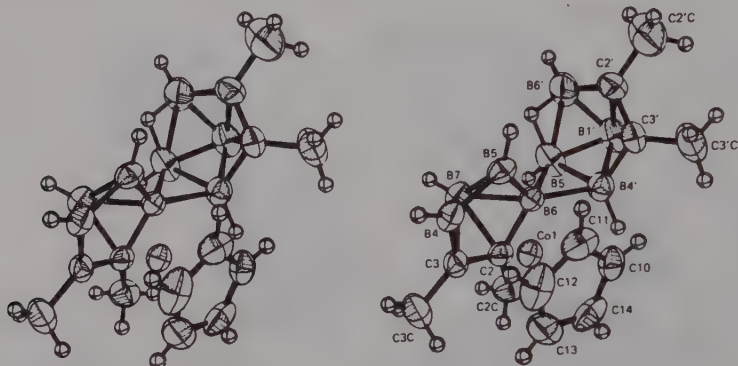


( $\eta^5$ -CYCLOPENTADIENYL) [ $\sigma^5$ -OCTAHYDROTETRAKIS(METHYLCARBA) DODECABORATO] COBALT  
 $C_{13}H_{25}B_8Co$

A.J. BORELLI, Jr., J.S. PLOTKIN and L.G. SNEDDON, 1982. *Inorg. Chem.*, **21**, 1328-1331.

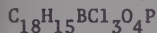
Monoclinic,  $P2_1/n$ ,  $a = 14.092$ ,  $b = 13.270$ ,  $c = 9.988$  Å,  $\beta = 103.06^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.042$  for 2282 reflexions.

The compound (Fig. 1) was shown to be a coupled-cage cobaltacarborane consisting of a  $[(CH_3)_2C_2B_4H_3]Co(\eta^5-C_5H_5)$  sandwich complex (Co-C 2.016(3)-2.049(3), Co-B 2.084(3)-2.124(3) Å) linked to a  $(CH_3)_2C_2B_4H_5$  carborane fragment by means of a 3-centre B-B-B bond (B-B 1.676(4), 1.842(4) and 1.978(4) Å).



Each structure contains tetrahedrally coordinated boron atoms, both the acetylacetonato and tropolonato moieties being delocalized and symmetric. The chelate rings in (Ia) and (Ib) have distorted B-envelope conformations; the chelate ring in II is essentially planar (Fig. 1). Mean B-O and B-C distances are: (Ia) 1.543(4), 1.605(4); (Ib) 1.533(3), 1.607(4); (II) 1.555(11), 1.605(6) Å.

## TRIPHENYL PHOSPHATE - BORON TRICHLORIDE ADDUCT



M.L. LEVIN, J.W. FIELDHOUSE and H.R. ALLCOCK, 1982. Acta Cryst., B38, 2284-2286.

Monoclinic,  $P2_1/c$ ,  $a = 9.194$ ,  $b = 20.293$ ,  $c = 11.488$  Å,  $\beta = 108.81^\circ$ ,  $D_m = 1.40$ ,  $Z = 4$ . Mo radiation,  $R = 0.041$  for 1637 reflexions.

In the adduct (Fig. 1) there is coordination between the phosphoryl O and the B atom, with B-O 1.511(5) Å. C-C bond distances are between 1.348(5) and 1.392(6) Å and bond angles within the planar phenyl rings  $117.39(4)$  to  $123.13(4)^\circ$ .

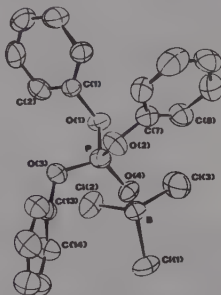


Fig. 1.  $\text{C}_{18}\text{H}_{15}\text{O}_4\text{P} \cdot \text{BCl}_3$ : perspective view of the adduct.

## TRIPHENYLPHOSPHINE BORANE

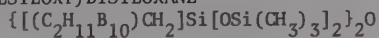
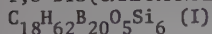


J.C. HUFFMAN, W.A. SKUPINSKI and K.G. CAULTON, 1982. Cryst. Struct. Comm., 11, 1435-1440.

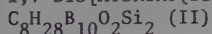
Triclinic,  $P\bar{1}$ ,  $a = 11.021$ ,  $b = 14.922$ ,  $c = 9.554$  Å,  $\alpha = 99.28$ ,  $\beta = 101.88$ ,  $\gamma = 89.31^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.082$  for 2360 reflexions (at  $-165^\circ\text{C}$ ).

The rotational conformation about the P-B bond in the two independent molecules in the asymmetric unit is staggered. Main dimensions are: mean P-B 1.917 Å, average H-B-H  $115^\circ$ , P-B-H  $103^\circ$ .

## 1,3-BIS(CARBORANYLMETHYL)-1,1,3,3-TETRAKIS(TRIMETHYLSILOXY)DISILOXANE



## 1,7-BIS[HYDROXY(DIMETHYL)SILYLMETHYL]-m-CARBORANE



A.I. YANOVSKII, I.L. DUBCHAK, V.E. SHKLOVER, Yu.T. STRUCHKOV, V.N. KALININ, B.A. IZMAILOV, V.D. MYAKUSHEV and L.I. ZAKHARKIN, 1982. Zh. Strukt. Khim., 23-5, 88-97 [J. Struct. Chem., 23, 728-736].

I. Monoclinic,  $P2_1/n$ ,  $a = 10.521$ ,  $b = 20.31$ ,  $c = 11.270$  Å,  $\beta = 107.45^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.045$  for 3036 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 7.131$ ,  $b = 11.47$ ,  $c = 12.26$  Å,  $\alpha = 108.33$ ,  $\beta = 97.11$ ,  $\gamma = 94.33^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.040$  for 2817 reflexions.

The two molecules are shown in Fig. 1. Molecule I is centrosymmetric with mean bond lengths: B-B 1.756(14), B-C 1.710(10), C-C(carborane) 1.664(4), and Si-C(H<sub>2</sub>) 1.863(3) Å. Molecule II has approximate  $C_{2v}$  symmetry with mean bond lengths: B-B 1.768(8), B-C 1.717(7), Si-C(H<sub>2</sub>) 1.884(4), Si-Me 1.848(3) Å. Other bond lengths in both molecules are as expected.

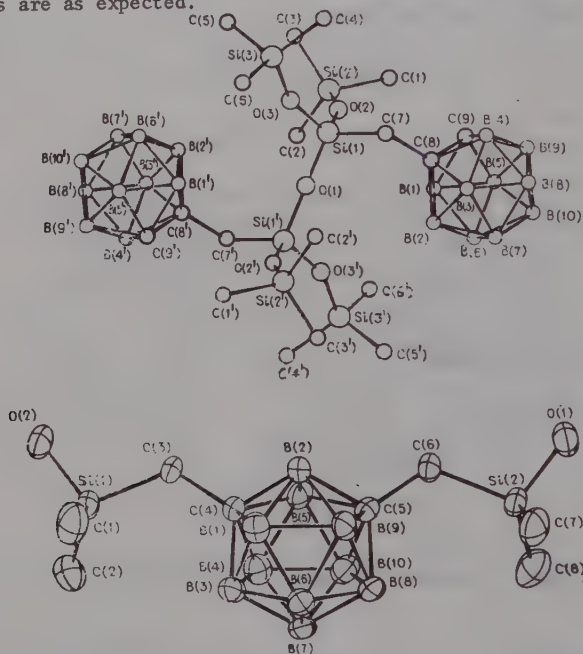


Fig. 1. The molecules  $\{[(C_2H_{11}B_{10})CH_2]Si[OSi(CH_3)_3]_2O$  (top) and  $[(CH_3)_2(HO)SiCH_2]_2(C_2H_{10}B_{10})$  (bottom).

BIS(*n*-BUTYLBORYL)-2-(*N,N'*-DICYCLOHEXYLAMIDINO)AMINOTHIAZOLATE  
 $C_{24}H_{43}BN_4S$

I.A. TESLYA, Z.A. STARIKOVA, V.A. DOROKHOV and B.M. MIKHAILOV, 1982. *Koord. Khim.*, 8, 1546-1549 [*Sov. J. Coord. Chem.*, 8, 847-851].

Orthorhombic,  $Fdd2$ ,  $a = 20.687$ ,  $b = 56.993$ ,  $c = 8.940$  Å,  $Z = 16$ . Mo radiation,  $R = 0.034$  for 1723 reflexions.

The bicyclic moiety (Fig. 1) is as a whole puckered, ring A being planar and forming an angle of  $3.8^\circ$  with the mean plane of chelate ring B. The configuration at N(3) is planar and that at N(1) and N(4) pyramidal. Cyclohexane rings C and D have chair conformations and are oriented perpendicularly to the bicyclic moiety. The N atoms are in equatorial positions. The boron atom has distorted tetrahedral geometry. The mean B-N and C-N distances in the chelate are 1.587 and 1.334 Å respectively. The mean C-S distance in thiazole ring A is 1.732 Å.

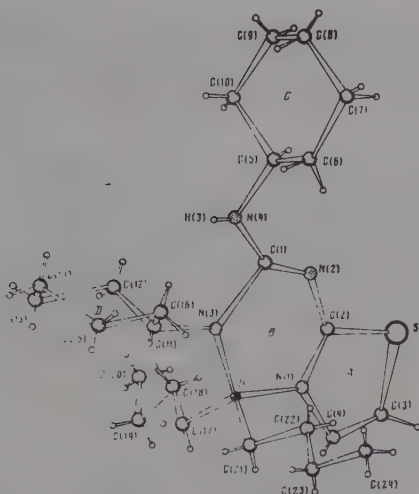


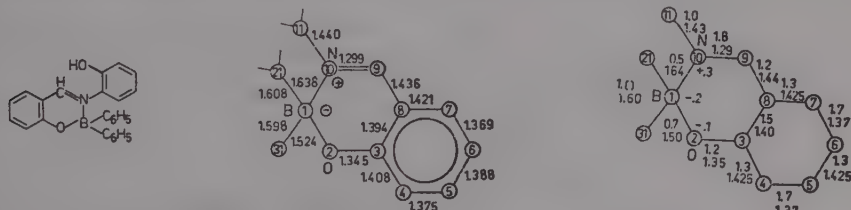
Fig. 1.  $C_{24}H_{43}BN_4S$ : the molecular structure.

3-(2-HYDROXYPHENYL)-2,2-DIPHENYL-1-OXA-3-AZONIA-2-BORATANAPHTHALENE METHANOL SOLVATE  
 $C_{25}H_{20}BNO_2,CH_4O$

R. ALLMANN, E. HOHAUS and S. OLEJNIK, 1982. *Z. Naturforsch.*, **37b**, 1450-1455.

Orthorhombic, Pbnb,  $a = 8.815$ ,  $b = 17.309$ ,  $c = 28.992$  Å,  $D_m = 1.226$ ,  $Z = 8$ . Mo radiation,  $R = 0.055$  for 1910 reflexions.

The heterocycle exhibits a half-chair conformation with the B atom 0.68 Å out of the best plane through the remaining 5 atoms. The bond lengths (Fig. 1) indicate some contribution of the O-chinoic form of ring C(3)-C(8). The molecules and solvate molecules are linked by hydrogen bonds (O-H...O 2.658(4), 2.770(4) Å).



1.363, B-C(1) 1.567 and B-C(11) 1.540 Å, with O-B-C(11) 117.1, O-B-C(1) 114.3 and C(1)-B-C(11) 128.6°. The C-SiMe<sub>3</sub> bonds (mean 1.900 Å) are probably longer than the Si-Me bonds (mean 1.867 Å), and the C-Si-Me angles (mean 113.0°) are larger than the Me-Si-Me angles (mean 105.6°). Atoms C(17) and C(18) are disordered.

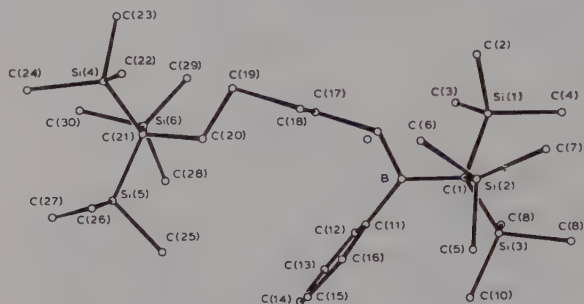


Fig. 1. Molecular structure of C<sub>30</sub>H<sub>67</sub>BOSi<sub>6</sub>.

SILYL MONOTHIOACETATE  
C<sub>2</sub>H<sub>6</sub>OSSi

M.J. BARROW, E.A.V. EBSWORTH, C.M. HUNTLEY and D.W.H. RANKIN, 1982. J. Chem. Soc. Dalton, 1131-1136.

Monoclinic, P2<sub>1</sub>/n, a = 5.40, b = 8.77, c = 11.47 Å, β = 96.0°, Z = 4. Cu radiation, R = 0.032 for 739 reflexions (at 130 K).

The analysis confirms that the silyl group is bonded through oxygen with Si-O 1.699(5) Å. The corresponding distance in the gas phase is 1.717(6) Å. The Si and S atoms (Fig. 1) are in the planar, cis conformation for both phases (Si...S 3.19 (3.14 Å gas)). The heavy atom skeleton deviates slightly but significantly from planarity in the crystal. The intermolecular S...Si distance is 3.382(10) Å. The O(2)-C(3), C(3)-S(4) and C(3)-C(5) distances in the molecule are 1.319(3), 1.627(3) and 1.484(4) Å respectively.

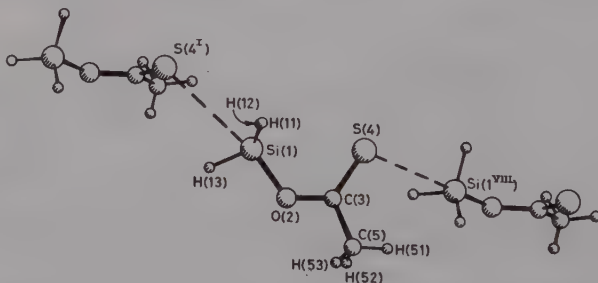


Fig. 1. C<sub>2</sub>H<sub>6</sub>OSSi: a view of the molecule and two of its neighbours.

SODIUM TRIMETHYLSILANOATE TRIHYDRATE  
C<sub>3</sub>H<sub>9</sub>NaOSi·3H<sub>2</sub>O (I)

DISODIUM TETRAMETHYLDISILOXANE-1,3-DIOLATE TETRAHYDRATE  
C<sub>4</sub>H<sub>12</sub>Na<sub>2</sub>O<sub>5</sub>Si<sub>2</sub>·4H<sub>2</sub>O (II)

I.L. DUBCHAK, V.E. SHKLOVER, M.Yu. ANTIPIN, Yu.T. STRUCHKOV, V.M. KOPILOV, A.M. MUZAFAROV, P.L. PRIKHOD'KO and A.A. ZHDANOV, 1982. Zh. Strukt. Khim., 23-2, 63-71 [J. Struct. Chem., 23, 219-227].

I. Monoclinic,  $C2/c$ ,  $a = 23.51$ ,  $b = 6.262$ ,  $c = 11.665$  Å,  $\beta = 91.12^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.072$  for 1413 reflexions (at  $-120^\circ\text{C}$ ).

II. Monoclinic,  $P2_1/c$ ,  $a = 8.967$ ,  $b = 24.459$ ,  $c = 12.142$  Å,  $\beta = 90.70^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.081$  for 2723 reflexions (at  $-120^\circ\text{C}$ ).

Both structures consist of double layers, each containing both hydrophilic and hydrophobic parts. The hydrophilic part consists of  $\text{Si-O}^-$  groups turned toward one another, with  $\text{Na}^+$  ions and water molecules between them. The hydrophobic part contains Me groups interacting via van der Waals forces. The  $\text{Na}^+$  ion in I is coordinated by five O atoms ( $\text{Na-O}$  2.316(4)-2.383(4) Å). The four independent  $\text{Na}^+$  ions in II are coordinated differently: two are five-coordinate ( $\text{Na-O}$  2.299(6)-2.491(6) Å), one is tetrahedrally coordinated ( $\text{Na-O}$  2.296(6)-2.438(7) Å), and the other is six-coordinate ( $\text{Na-O}$  2.302(8)-2.606(6) Å). Anion geometry is normal. The  $[\text{O}(\text{Me})_2\text{Si}]_2\text{O}^{2-}$  anion in II is shown in Fig. 1.

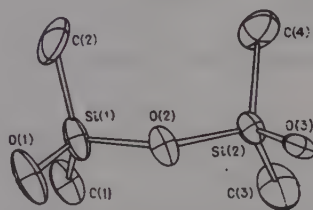
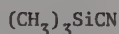


Fig. 1. The  $[\text{O}(\text{Me})_2\text{Si}]_2\text{O}^{2-}$  anion in  $\text{C}_4\text{H}_{12}\text{Na}_2\text{O}_3\text{Si}_2 \cdot 4\text{H}_2\text{O}$ . One of the two independent species is shown.

#### TRIMETHYLSILYL CYANIDE

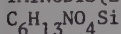


M.J. BARROW, 1982. *Acta Cryst.*, **B38**, 150-154.

Orthorhombic,  $\text{Pnm}2_1$ ,  $a = 6.62$ ,  $b = 7.45$ ,  $c = 6.69$  Å (at 140K),  $Z = 2$ . Cu radiation,  $R = 0.082$  for 224 reflexions (photographic data).

The structure, which is disordered, consists of chains of molecules aligned head-to-tail with an intermolecular  $\text{N} \cdots \text{Si}$  distance of 3.66 Å. The disorder seems to indicate that the chains may be aligned either parallel or antiparallel to  $c$ . A composite structure, which attempts to model the disorder, gives the above  $R$ -factor.

#### IMINO BIS(ETHYLENEOXO)ETHYLENEDIOXOSILANE



D. SCHOMBURG, 1982. *Z. Anorg. Chem.*, **493**, 53-58.

Monoclinic,  $Cc$ ,  $a = 15.563$ ,  $b = 6.741$ ,  $c = 10.630$  Å,  $\beta = 130.29^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.033$  for 1338 reflexions.

The Si atom (Fig. 1) is trigonal-bipyramidally coordinated with Si-N 2.004(3), Si-O(1) 1.665(3), Si-O(2) 1.667(2), Si-O(3) 1.710(2), Si-O(4) 1.683(3) Å. The three five-membered chelate rings each adopt the envelope conformation with C(2), C(3) and C(5) at the flaps. Molecules are linked by  $\text{N-H} \cdots \text{O}$  hydrogen bonds (2.93(1) Å).



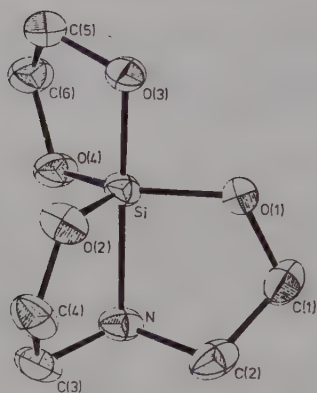


Fig. 1. The molecular structure of  $C_6H_{13}NO_4Si$ .

# CYCLOHEXYLSILANETRIOL

$C_6H_{14}O_3Si$

$C_6H_{11}Si(OH)_3$

H. ISHIDA, J.L. KOENIG and K.C. GARDINER, 1982. J. Chem. Phys., 77, 5748-5751.

Monoclinic,  $C2/m$ ,  $a = 7.876$ ,  $b = 6.637$ ,  $c = 15.802$  Å,  $\beta = 95.51^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.057$  for 1031 reflexions (at  $-60^\circ C$ ).

The molecules (Fig. 1) lie on a crystallographic mirror plane and they pack in a head-to-head, tail-to-tail arrangement with the cyclohexyl groups forming a hydrophobic double sheet and the silanetriol groups forming a second, hydrophilic, double sheet. The structure has a distorted hydrogen bond network with the hydrogen on each oxygen having equal probability of hydrogen bonding to oxygens in two different molecules. Two types of hydrogen bonds exist in the hydrophilic double sheet;  $O(1) \dots O(2)$  intrasheet hydrogen bonds,  $2.724(2)$  Å, and  $O(2) \dots O(2)$  intrasheet hydrogen bond,  $2.722(3)$  Å. Some other dimensions are  $Si-O(1)$   $1.637(2)$ ,  $Si-O(2)$   $1.608(2)$  Å,  $O(1)-Si-O(2)$   $105.94(8)$ ,  $O(2)-Si-O(2)$   $112.8(2)^\circ$ .

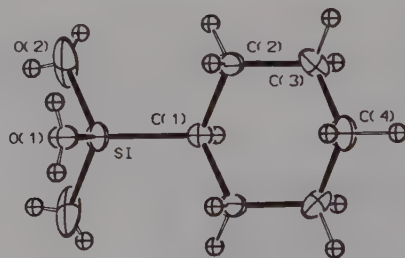


Fig. 1. The cyclohexylsilanetriol molecule.

# SODIUM 3-(TRIMETHYLSILYL)-1-PROPANE-SULPHONATE HYDRATE

$C_6H_{15}NaO_3SSi \cdot H_2O$

$C_6H_{15}O_3SSi^-Na^+ \cdot H_2O$

B. TINANT, B. COENE, J.P. DECLERCQ, G. GERMAIN and M. VAN MEERSSCHE, 1982. Bull. Soc. Chim. Belg., 91, 95-96.

Triclinic,  $P\bar{1}$ ,  $a = 5.914$ ,  $b = 10.816$ ,  $c = 18.869$  Å,  $\alpha = 97.96$ ,  $\beta = 95.46$ ,  $\gamma = 89.76^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.047$  for 2319 reflexions.

The two independent formula units in the asymmetric unit are almost identical (Fig. 1). The mean Si-C distance is 1.856(6) Å. Each Na<sup>+</sup> cation is surrounded by six oxygen atoms at 2.30-2.50 Å; two oxygens are from water molecules and the others are sulphonate oxygens.

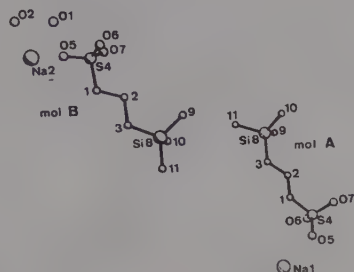


Fig. 1. The two formula units of  $C_6H_{15}O_3SSi^-Na^+, H_2O$ .

1,1-DIMETHYL-2,5-BIS(CHLORODIMETHYLSILYL)-1-SILA-2,5-DIAZACYCLOPENTANE-3,4-DITHIONE  
 $C_8H_{18}Cl_2N_2S_2Si_3$

H.W. ROESKY, H. HOFMANN, W. CLEGG, M. NOLTEMEYER and G.M. SHELDRICK, 1982. Inorg. Chem., 21, 3798-3800.

Orthorhombic, Cmc<sub>2</sub>m,  $a = 10.390$ ,  $b = 8.107$ ,  $c = 21.387$  Å,  $Z = 4$ . Mo radiation,  $R = 0.053$  for 706 reflexions.

The molecule (Fig. 1) possesses exact mm symmetry, with all atoms except those of the methyl groups coplanar. Bond lengths: S-C 1.637(5), Si-C1 2.075(2), Si(1)-N 1.770(4), Si(2)-N 1.784(4), Si-C 1.832(5) and 1.839(5), C-N 1.362(6), C-C 1.535(8) Å.

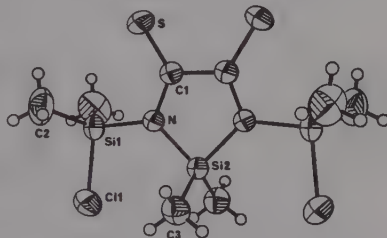


Fig. 1. The  $C_8H_{18}Cl_2N_2S_2Si_3$  molecule.

trans-2,4-BIS(TRIMETHYLSILYL)-2,4-DIHYDRO-1,3-DITHIETANE-1,1,3,3-TETROXIDE  
 $C_8H_{20}O_4S_2Si_2$  (I)

2,2,4,4-TETRAMETHYL-3-PYRROLIDINOCYCLOBUTANONE  
 $C_{12}H_{21}NO$  (II)

2,2,4,4-TETRAMETHYL-1-PHENYL-3-PYRROLIDINOCYCLOBUTANOL  
 $C_{18}H_{27}NO$  (III)

B. BALBACH, R. ALT and M.L. ZIEGLER, 1982. Z. Naturforsch., 37b, 1005-1013.

I. Rhombohedral,  $R\bar{3}$ ,  $a = 12.722$ ,  $c = 26.127$  Å,  $D_m = 1.24$ ,  $Z = 9$ . Mo radiation,  $R = 0.025$  for 909 reflexions.

II. Monoclinic,  $P2_1/n$ ,  $a = 6.041$ ,  $b = 13.071$ ,  $c = 15.589$  Å,  $\beta = 99.32^\circ$ ,  $D_m = 1.00$ ,  $Z = 4$ . Mo radiation,  $R = 0.041$  for 763 reflexions.

III. Monoclinic,  $P2_1/n$ ,  $a = 14.953$ ,  $b = 15.867$ ,  $c = 15.457$  Å,  $\beta = 116.15^\circ$ ,  $D_m = 1.14$ ,  $Z = 8$ . Mo radiation,  $R = 0.051$  for 2123 reflexions.

In I (Fig. 1) the  $C_2S_2$  ring is planar and its substituents are in trans positions. The plane of the four O atoms is perpendicular (dihedral angle  $90.1(3)^\circ$ ) to the  $C_2S_2$  ring. In II the four-membered ring is folded about the  $C(1)\dots C(4)$  line (dihedral angle between planes  $C(1)C(3)C(4)$  and  $C(1)C(2)C(4)$  is  $18.4(4)^\circ$ ). A similar folding is also found in III where the mean dihedral angle between planes  $C(11)C(12)C(13)$  and  $C(11)C(14)C(13)$  is  $27.1(4)^\circ$ . Bond lengths and angles have expected values.

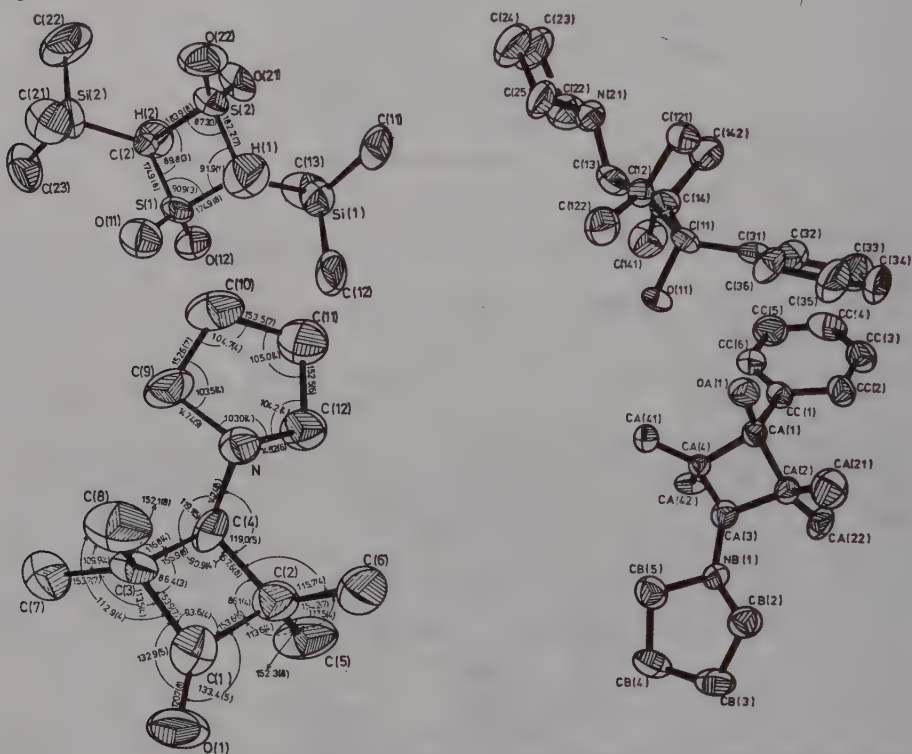


Fig. 1. Bond lengths (pm) and angles for  $C_6H_{20}O_4S_2Si_2$  (I) (top left) and for  $C_{12}H_{21}NO$  (II) (lower left), and the two independent molecules of  $C_{18}H_{27}NO$  (III) (right).

3,5-DIAMINO-2,2,6,6-TETRAMETHYL-4-PHENOXY-1-OXA-3,5-DIAZA-4-PHOSPHA-2,6-DISILACYCLOHEXANE 4-SULPHIDE

$C_{10}H_{21}N_4O_2PSSi_2$

U. ENGELHARDT, T. BÜNGER and B. STROMBURG, 1982. Acta Cryst., B38, 1173-1176.

Monoclinic,  $P2_1/c$ ,  $a = 14.784$ ,  $b = 13.880$ ,  $c = 9.581$  Å,  $\beta = 108.80^\circ$ ,  $D_m = 1.24$ ,  $Z = 4$ . Cu radiation,  $R = 0.044$  for 2580 reflexions.

In the molecule (Fig. 1) the six-membered heterocycle has a slightly twisted, almost planar conformation. Rather large P-N-Si ( $134.9$  and  $133.8^\circ$ ) and Si-O-Si ( $135.8^\circ$ ) angles are observed in the ring. The exocyclic N-N distances, mean value  $1.458$  Å, are somewhat longer than in other thiophosphoric acid hydrazides.

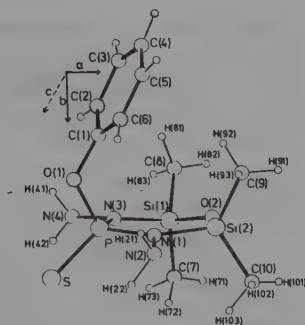


Fig. 1.  $C_{10}H_{21}N_4O_2PSSi_2$ : a perspective view of the molecule.

HEXAMETHYLENEDIAMMONIUM BIS(ETHYLENEDIOXY)METHYLSILANE  
 $C_{11}H_{29}N_2O_4Si$

$C_6H_{18}N_2^{2+}(C_2H_4O_2)_2Si(CH_3)^{2-}$

D. SCHOMBURG, 1982. *Z. Naturforsch.*, **37b**, 195-197.

Hexagonal,  $P6_122$ ,  $a = 11.902$ ,  $c = 30.148$  Å,  $Z = 6$ . Mo radiation,  $R = 0.041$  for 1977 reflexions.

In the anion (Fig. 1) the Si atom coordination geometry is between trigonal-bipyramidal and square-pyramidal. Principal bond lengths and angles are: Si-C 1.876(3), Si-O(1) 1.769(2), Si-O(2) 1.749(2), Si-O(3) 1.711(2), Si-O(4) 1.735(2) Å; O(1)-Si-O(3) 88.3(1), O(1)-Si-O(4) 87.9(1), O(1)-Si-O(2) 161.7(1), O(2)-Si-O(3) 86.5(1), O(2)-Si-O(4) 87.9(1), O(3)-Si-O(4) 136.0(1), C-Si-O 98.6(2)-112.8(2)°. The cations and anions are linked by N-H...O hydrogen bonds (2.744(3)-2.834(3) Å).

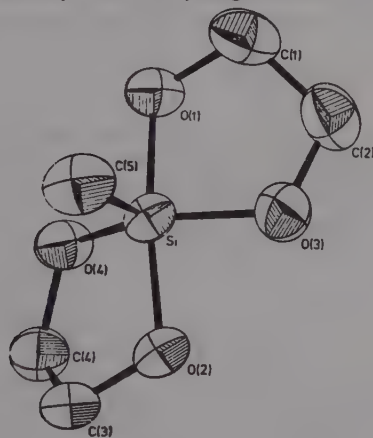


Fig. 1. The  $(C_2H_4O_2)_2Si(CH_3)^{2-}$  anion.

1-m-CHLOROPHENOXY-SILATRANE  
 $C_{12}H_{16}ClNO_4Si$

L. PÁRKÁNYI, P. HENCSEI and L. BIHÁTSI, 1982. *J. Organometal. Chem.*, **232**, 315-323.

Monoclinic,  $P2_1/c$ ,  $a = 11.406$ ,  $b = 8.017$ ,  $c = 14.455$  Å,  $\beta = 97.20^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.041$  for 2382 reflexions.

The pentavalent Si atom is coordinated by four O atoms with a mean Si-O(equatorial) distance of 1.656 Å (Fig. 1). The Si-O(12) distance is 1.690 Å. The trans-annular N→Si dative bond is rather short (2.079 Å) and the crystal structure is partially disordered with C atoms attached to N appearing on both sides of the Si,O(eq.),C,N plane with a population of 0.8 and 0.2 respectively. The m-chlorophenoxy group forms a dihedral angle of -57.4° with the plane of O(2),O(8),O(9).

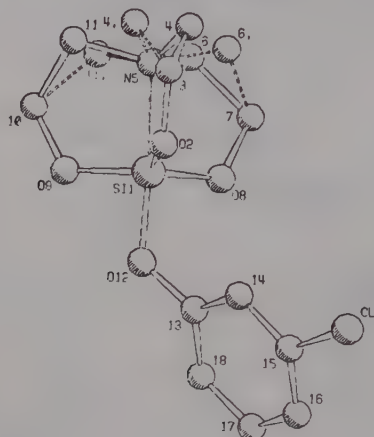


Fig. 1. Molecular structure of 1-m-chlorophoxysilatrane. C atoms marked with a comma have multiplicity 0.2.

4,5-DI-*t*-BUTYL-1,1,2,2-TETRAFLUORO-1,2-DISILACYCLOHEXA-3,5-DIENE  
 $C_{12}H_{20}F_4Si_2$

T.-H. HSEU, Y. CHI and C.-S. LIU, 1982. J. Am. Chem. Soc., 104, 1594-1598.

Monoclinic,  $P2_1/c$ ,  $a = 12.584$ ,  $b = 16.843$ ,  $c = 15.293$  Å,  $\beta = 69.70^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.083$  for 1923 reflexions.

Each of the two crystallographically independent molecules adopt a twisted-boat conformation which deviates somewhat from  $C_2$  symmetry. The bonds connecting the two tert-butyl groups are long, averaging 1.54(2) Å, as compared to expected value of 1.48 Å. The averaged Si-Si bond length and Si-Si-C bond angles are 2.331(7) Å and  $95.5^\circ$ . The C=C bonds are all twisted, averaged C=C lengths and twist angles are 1.345 Å for a twist of  $24^\circ$  and 1.388 Å for a twist of  $27^\circ$ .

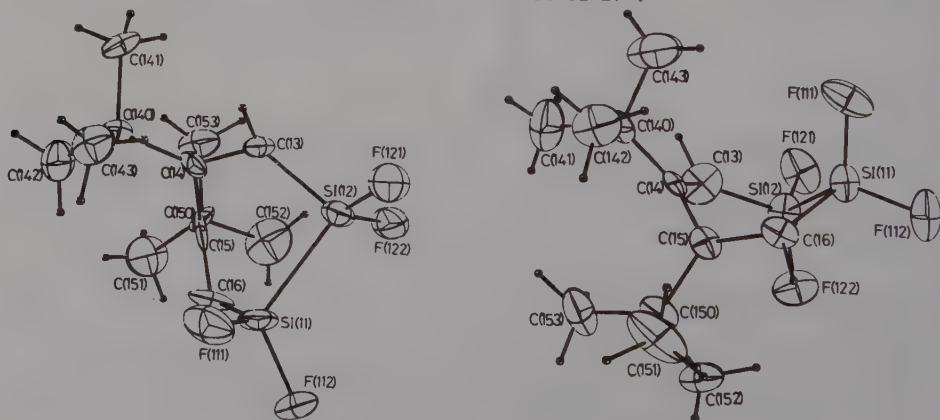


Fig. 1. Two views of the  $C_{12}H_{20}F_4Si_2$  molecule.

2,8-DISILA-2,2,8,8-TETRAMETHYLTRICYCLO[7.3.0.0<sup>3</sup>,<sup>7</sup>]DODECA-3,5,9,11-TETRAENE  
 $C_{14}H_{20}Si_2$

V.K. BELSKY, N.N. ZEMLYANSKY, I.V. BORISOVA, N.D. KOLOSOVA and I.P. BELETSKAYA, 1982.  
 Cryst. Struct. Comm., 11, 497-500.

Monoclinic,  $P2_1/n$ ,  $a = 8.823$ ,  $b = 9.258$ ,  $c = 9.040$  Å,  $\beta = 107.82^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.028$  for 1061 reflexions (at 120 K).

The molecule lies about an inversion centre; the six-membered ring has a chair conformation with the Si atoms displaced by 0.852 Å on opposite sides of the plane of the C atoms. The five-membered rings have a slight envelope conformation, with C(2) at the flap (flap angle  $4.8^\circ$ ). The Si-C distances are: Si-C(1) 1.858(2), Si-C(6) 1.867(2), Si-C(7) 1.863(2), Si-C(2') 1.918(3) Å. Bond lengths in the five-membered ring are: C(1)-C(2) 1.502(3), C(2)-C(3) 1.481(3), C(3)-C(4) 1.347(3), C(4)-C(5) 1.452(3), C(1)-C(5) 1.358(3) Å.

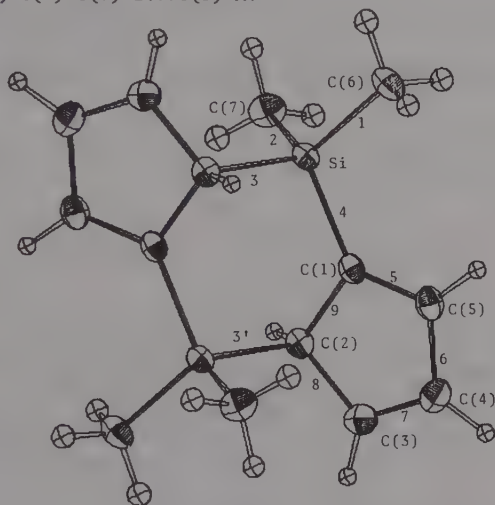
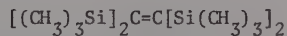


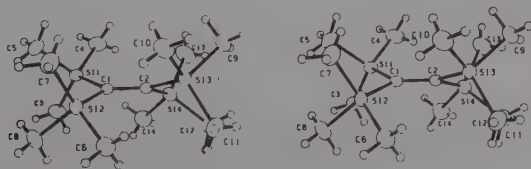
Fig. 1. A view of the  $C_{14}H_{20}Si_2$  molecule.

TETRAKIS(TRIMETHYLSILYL)ETHYLENE  
 $C_{14}H_{36}Si_4$



H. SAKURAI, Y. NAKADAIRA, H. TOBITA, T. ITO, K. TORIUMI and H. ITO, 1982. J. Am. Chem. Soc., 104, 300-302.

Monoclinic,  $P2_1/n$ ,  $a = 17.841$ ,  $b = 12.432$ ,  $c = 9.598$  Å,  $\beta = 92.88^\circ$ ,  $D_m = 0.961$ ,  $Z = 4$ . Mo radiation,  $R = 0.045$  for 4049 reflexions ( $D_m$  at  $23.5^\circ C$ , other data at  $-70^\circ C$ ).





The molecule (Fig. 1) contains a long C=C double bond (1.368(3) Å). Although each of C(1) and C(2) display planar coordination, the dihedral angle between these planes is 29.5°. The mean Si-C(sp<sup>3</sup>) distance of 1.871(8) Å is normal while the mean Si-C(sp<sup>2</sup>) distance of 1.915(4) Å is unusually long as a result of the overcrowding.

N,N'-(3,3,5,5-TETRAMETHYL-3,5-DISILAHEPTAMETHYLENE) BIS(TRIMETHYLAMMONIUM) DIIODIDE  
 $C_{15}H_{40}I_2N_2Si_2$  (I)  $C_{15}H_{40}N_2Si_2^{2+}, 2I^-$

N,N'-(3,3,6,6-TETRAMETHYL-3,6-DISILAOCTAMETHYLENE) BIS(TRIMETHYLAMMONIUM) DIIODIDE  
 $C_{16}H_{42}I_2N_2Si_2$  (II)  $C_{16}H_{42}N_2Si_2^{2+}, 2I^-$

N,N'-(3,3,7,7-TETRAMETHYL-3,7-DISILANONAMETHYLENE) BIS(TRIMETHYLAMMONIUM) DIIODIDE  
 $C_{17}H_{44}I_2N_2Si_2$  (III)  $C_{17}H_{44}N_2Si_2^{2+}, 2I^-$

N,N'-(3,3,8,8-TETRAMETHYL-3,8-DISILADECAMETHYLENE) BIS(TRIMETHYLAMMONIUM) DIIODIDE  
 $C_{18}H_{46}I_2N_2Si_2$  (IV)  $C_{18}H_{46}N_2Si_2^{2+}, 2I^-$

N,N'-(3,3,9,9-TETRAMETHYL-3,9-DISILAUNDECAMETHYLENE) BIS(TRIMETHYLAMMONIUM) DIIODIDE  
 $C_{19}H_{48}I_2N_2Si_2$  (V)  $C_{19}H_{48}N_2Si_2^{2+}, 2I^-$

R. TACKE, H. LINO, M.T. ATTAR-BASHI, W.S. SHELDRIK, L. ERNST, R. NIEDNER and J. FROHNECKE, 1982. Z. Naturforsch., 37b, 1461-1471.

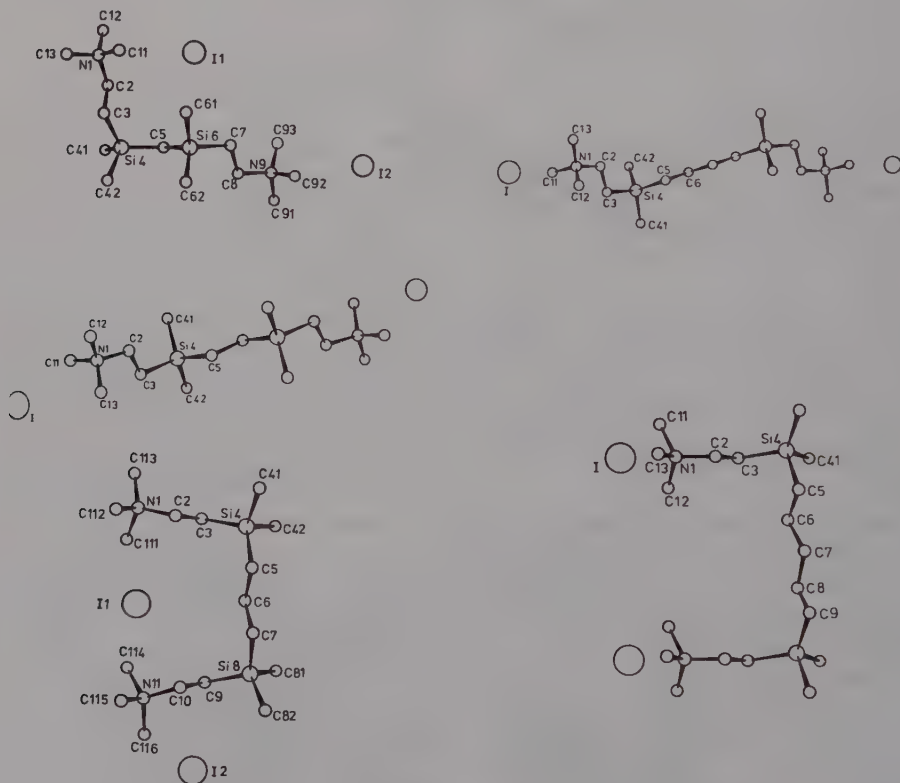


Fig. 1. Perspective views of the trimethylammonium diiodide derivatives (I) (top left), (II) (centre left), (III) (bottom left), (IV) (top right) and (V) (bottom right).

I. Monoclinic,  $P2_1/c$ ,  $a = 14.341$ ,  $b = 13.800$ ,  $c = 13.079$  Å,  $\beta = 100.52^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.040$  for 3554 reflexions.

II. Monoclinic,  $P2_1/n$ ,  $a = 8.940$ ,  $b = 15.416$ ,  $c = 9.711$  Å,  $\beta = 97.50^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.058$  for 2331 reflexions.

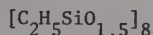
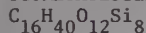
III. Monoclinic,  $P2_1/c$ ,  $a = 6.411$ ,  $b = 26.586$ ,  $c = 17.060$  Å,  $\beta = 106.90^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.060$  for 2557 reflexions.

IV. Monoclinic,  $P2_1/c$ ,  $a = 9.257$ ,  $b = 10.957$ ,  $c = 14.073$  Å,  $\beta = 93.77^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.043$  for 2572 reflexions.

V. Monoclinic,  $P2_1$ ,  $a = 16.754$ ,  $b = 7.747$ ,  $c = 5.822$  Å,  $\beta = 93.13^\circ$ ,  $Z = 1$ . Mo radiation,  $R = 0.045$  for 1572 reflexions.

The bond lengths and angles in the five structures (Fig. 1) have normal values. Structures II and IV have crystallographic  $C_i$  symmetry. The molecular conformations are: I aggaag<sup>-</sup>a; II agaaag<sup>-</sup>a; III aggaag<sup>-</sup>g<sup>-</sup>a; IV agaaaaag<sup>-</sup>a; and V aggaaaag<sup>-</sup>g<sup>-</sup>a (where a = anti, g = gauche and g<sup>-</sup> = gauche with opposite sign).

#### OCTAETHYLSILASESIQUOXANE



N.V. PODBEREZSKAYA, S.A. MAGARILL, I.A. BAIDINA, S.V. BORISOV, L.É. GORSH, A.N. KANEV and T.N. MARTYNOVA, 1982. Zh. Strukt. Khim., 23-3, 120-129 [J. Struct. Chem., 23, 422-431].

Triclinic,  $P\bar{1}$ ,  $a = 9.433$ ,  $b = 9.433$ ,  $c = 9.433$  Å,  $\alpha = 95.92^\circ$ ,  $\beta = 95.92^\circ$ ,  $\gamma = 95.92^\circ$ ,  $D_m = 1.30$ ,  $Z = 1$ . Mo radiation,  $R = 0.12$  for 1249 reflexions. (The structure may also be considered as disordered in  $R\bar{3}$ ;  $a = 9.433$  Å,  $\alpha = 95.92^\circ$ .)

The structure (Fig. 1) consists of centrosymmetric molecules, the framework of which consists of six eight-membered  $Si_4O_4$  rings. Mean bond lengths: Si-O 1.61, Si-C 2.01 Å. Terminal ethyl carbons are threefold disordered.

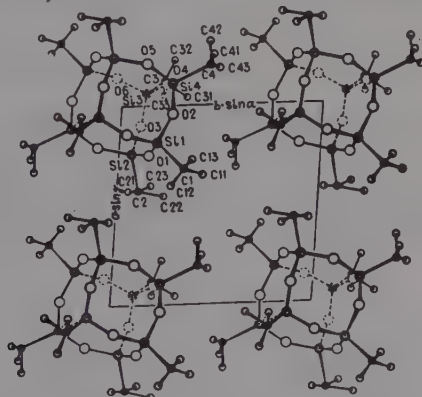


Fig. 1. The structure of  $C_{16}H_{40}O_{12}Si_8$ .

2,2-DIPHENYL-1-OXA-2-SILA-1,2-DIHYDRONAPHTHALENE  
 $C_{20}H_{16}OSi$  (I)

2,2-DICHLORO-3-TRICHLOROSILYL-1-OXA-2-SILA-1,2-DIHYDRONAPHTHALENE  
 $C_8H_5Cl_5OSi_2$  (II)

V.A. SHARAPOV, A.I. GUSEV, V.K. BEL'SKII, V.E. ZAVODNIK, V.V. STEPANOV, T.L. KRASNOVA, E.A. CHERNYSHEV and Yu.T. STRUCHKOV, 1982. Zh. Strukt. Khim., 23-1, 140-147 [J. Struct. Chem., 23, 115-121].

I. Monoclinic,  $P2_1/b$ ,  $a = 10.598$ ,  $b = 10.661$ ,  $c = 14.659$  Å,  $\gamma = 104.30^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.086$  for 1179 reflexions.

II. Orthorhombic,  $Pbam$ ,  $a = 24.760$ ,  $b = 15.705$ ,  $c = 7.126$  Å,  $Z = 8$ . Mo radiation,  $R = 0.155$  for 1100 reflexions.

The two molecules (Fig. 1) are characterized by a planar conformation of the fused-ring system, and short intracyclic Si-C distances of 1.838(5) Å in I and 1.82(2) Å in II. The structure II contains two independent and virtually identical molecules possessing exact mirror symmetry.

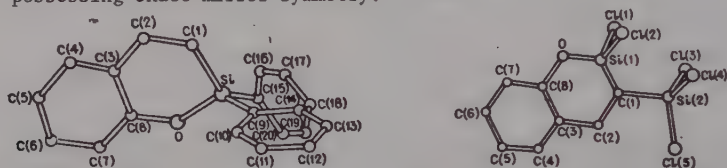


Fig. 1. The structures of the  $C_{20}H_{16}OSi$  (left) and  $C_8H_5Cl_5OSi_2$  (right) molecules.

1,1,3,3,5,5,7,7-OCTAMETHYL-2,6-BIS(PENTAFLUOROPHENYL)-CYCLO-1,3,5,7-TETRASILA-2,6-DIAZA-4,8-DIOXANE

$C_{20}H_{24}F_{10}N_2O_2Si_4$

$[(Me_2Si)_2ONC_6F_5]_2$

P. CLARE, D.B. SOWERBY and I. HAIDUC, 1982. J. Organometal. Chem., 236, 293-299.

Triclinic,  $P\bar{1}$ ,  $a = 8.703$ ,  $b = 14.334$ ,  $c = 13.021$  Å,  $\alpha = 102.05$ ,  $\beta = 107.43$ ,  $\gamma = 105.83^\circ$ ,  $D_m = 1.4$ ,  $Z = 2$ . Mo radiation,  $R = 0.039$  for 3928 reflexions.

The asymmetric unit consists of two half molecules, each of which lies on a crystallographic centre of symmetry. Each of the rings has a chair conformation (Fig. 1) with mean Si-N and Si-O distances of 1.747 and 1.623 Å respectively. The endocyclic angles at the Si atoms in the asymmetric unit are different (mean values 109.6 and 106.3°) and the ring angles at O and N have mean values of 147.7 and 123.2° respectively. The  $C_6F_5$  groups are normal with mean C-C 1.372 and mean C-F 1.339 Å.

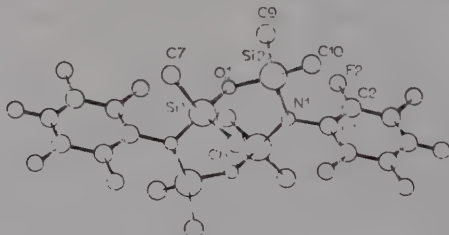


Fig. 1. Molecular structure of  $[(Me_2Si)_2ONC_6F_5]_2$  (molecule 1).

4-(2-(DIMETHYL-*t*-BUTYL-SILOXY)-3-HYDROXYMETHYL-3-METHYL-4,6,9-TRIOXADECYL)-5-HYDROXY-METHYL-TETRAHYDROFURAN-2-ONE

$C_{20}H_{40}O_8Si$

J.A. MARSHALL and R.D. ROYCE, Jr., 1982. *J. Org. Chem.*, **47**, 693-698.

Triclinic,  $P\bar{1}$ ,  $a = 11.065$ ,  $b = 12.665$ ,  $c = 10.381$  Å,  $\alpha = 109.91$ ,  $\beta = 96.53$ ,  $\gamma = 112.03^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.069$  for 2794 reflexions.

The X-ray analysis of this diol, Fig. 1, confirms the structure of the intermediate to be used in the synthesis of the cembranolide, crassin acetate.

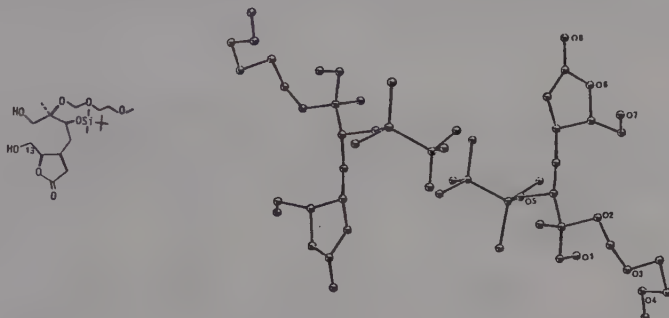


Fig. 1. Formula of the diol,  $C_{20}H_{40}O_8Si$ , and a view of a d,l pair of enantiomers as found in the crystal.

1,1-BIS(TRIMETHYLSILYL)-2-(TRIMETHYLSILOXY)-2-(1-ADAMANTYL)-1-SILAETHENE  
 $C_{20}H_{42}OSi_4$   $[(CH_3)_3Si]_2Si=C(C_{10}H_{15})[OSi(CH_3)_3]$

A.G. BROOK, S.C. NYBURG, F. ABDESAKEN, B. GUTEKUNST, G. GUTEKUNST, R.K.M.R. KALLURY, Y.C. POON, Y.-M. CHANG and W. WONG-NG, 1982. *J. Am. Chem. Soc.*, **104**, 5667-5672.

Triclinic,  $P\bar{1}$ ,  $a = 6.541$ ,  $b = 10.057$ ,  $c = 20.391$  Å,  $\alpha = 101.69$ ,  $\beta = 90.86$ ,  $\gamma = 105.77^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.049$  for 3067 reflexions (at  $-50^\circ C$ ).

The molecule is slightly twisted about the  $Si=C$  double bond ( $14.6^\circ$ ). Important structural parameters are shown in Fig. 1 (esd's  $< 0.005$  Å).

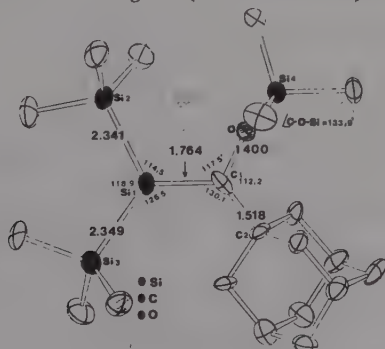


Fig. 1. The  $[(CH_3)_3Si]_2Si=C(C_{10}H_{15})[OSi(CH_3)_3]$  molecule.

1,2,4,5-TETRA-*t*-BUTYL-3,3,6,6-TETRAMETHYL-1,2,4,5-TETRAPHOSPHA-3,6-DISILACYCLOHEXANE  
 $C_{20}H_{48}P_4Si_2$   $\{(CH_3)_2Si[(CH_3)_3CP]_2\}_2$

R. FRÖHLICH and K.-F. TEBBE, 1982. *Acta Cryst.*, B38, 115-120.

Monoclinic,  $P2_1/n$ ,  $a = 9.170$ ,  $b = 27.716$ ,  $c = 11.840$  Å,  $\beta = 108.72^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.064$  for 2559 reflexions.

The molecule (Fig. 1) has a six-membered ring with the conformation of a nearly ideal boat. This conformation results from the repulsion of the all-trans substituted *t*-butyl groups and by the repulsion of the lone pairs on the P atoms. The geometry of the methyl and *t*-butyl substituents is quite normal. Some of the latter are slightly disordered.

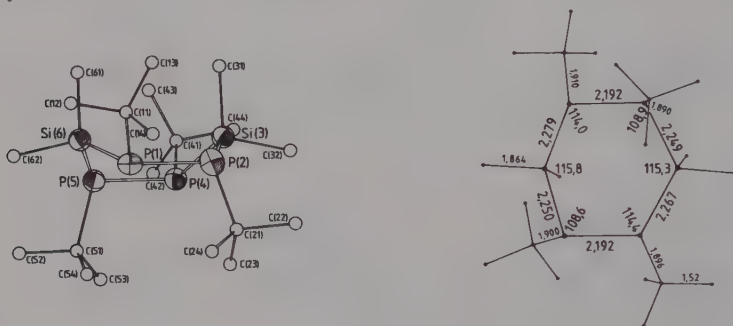


Fig. 1.  $C_{20}H_{48}P_4Si_2$ : perspective view of the molecule (left) and bond lengths and angles (right).

1,1-BIS(*t*-BUTYLDIMETHYLSILYL)-2,2-BIS(TRIMETHYLSILYL)ETHYLENE  
 $C_{20}H_{48}Si_4$   $[(t-C_4H_9)(CH_3)_2Si]_2C=C[Si(CH_3)_3]_2$

H. SAKURAI, H. TOBITA, Y. NAKADAIRA and C. KABUTO, 1982. *J. Am. Chem. Soc.*, 104, 4288-4289.

Monoclinic,  $C2/c$ ,  $a = 18.271$ ,  $b = 9.949$ ,  $c = 16.487$  Å,  $\beta = 117.60^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.067$  for 2170 reflexions.

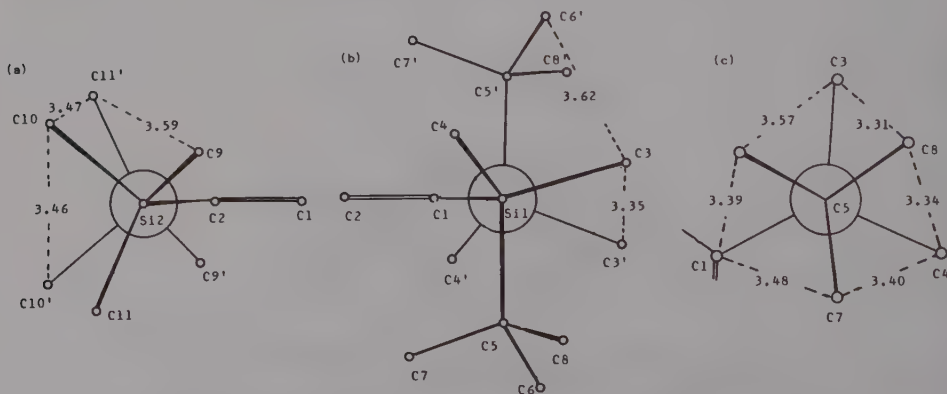


Fig. 1. Newman projections for  $[(t-C_4H_9)(CH_3)_2Si]_2C=C[Si(CH_3)_3]_2$ : (a) through Si(1) and Si(1'); (b) through Si(2) and Si(2'); (c) through Si(1) and C(5).

The molecule has crystallographic  $C_2$  symmetry. The C=C distance of 1.370 Å and the C(sp<sup>2</sup>)-Si distances of 1.915 and 1.926 Å are considerably longer than normal. As a result of steric crowding, the dihedral angle between the two C(sp<sup>2</sup>) planes is 49.6°. The conformations about the geminal Si atoms (shown in Fig. 1) are also of interest.

# 1,1,1-TRIMETHYLTRIPHENYLDISILANE

$C_{21}H_{24}Si_2$

$(CH_3)_3Si-Si(C_6H_5)_3$

L. PÁRKÁNYI and E. HENGGE, 1982. J. Organometal. Chem., 235, 273-276.

Trigonal,  $P\bar{3}$ ,  $a = 11.313$ ,  $c = 8.817$  Å,  $Z = 2$ . Mo radiation,  $R = 0.032$  for 1138 reflexions.

The molecule (Fig. 1) possesses threefold crystallographic symmetry and the relative positions of the methyl groups and phenyl rings can be characterized by the torsion angle C(Me)-Si-Si-C(Ph) of 34.2°. The Si(1)-Si(2) bond length is 2.355 Å and the Si(1)-C(1) and Si(2)-C(7) lengths are 1.886 and 1.862 Å respectively. Some angles are Si(2)-Si(1)-C(1) 110.3, Si(1)-Si(2)-C(7) 109.9, C(1)-Si(1)-C(1') 108.7 and C(7)-Si(2)-C(7') 109.0°.

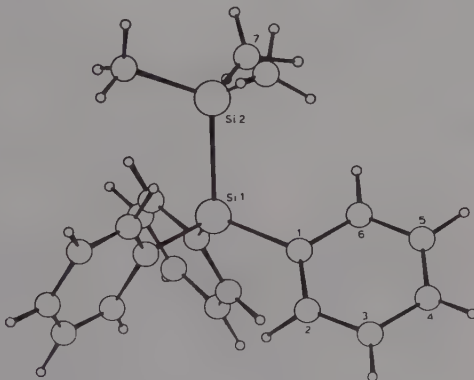
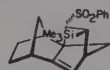


Fig. 1. Molecular structure of  $C_{21}H_{24}Si_2$ .

# exo-2-(PHENYLSULFONYL)-endo-3-(TRIMETHYLSILYL)-anti-TETRACYCLO[4.4.0.1<sup>1,4</sup>.1<sup>7,10</sup>]-DODEC-5-ENE

$C_{21}H_{28}O_2SSi$



L.A. PAQUETTE, R.V. WILLIAMS, R.V.C. CARR, P. CHARUMILIND and J.F. BLOUNT, 1982. J. Org. Chem., 47, 4566-4572.

Monoclinic,  $P2_1/a$ ,  $a = 12.749$ ,  $b = 15.586$ ,  $c = 10.969$  Å,  $\beta = 113.73^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.038$  for 1636 reflexions.

The analysis established the structure as shown above.



2',3'-O-(TETRAISOPROPYL-1,3-DISILOXANEDIYL)CYTIDINE  
 $C_{21}H_{39}N_3O_6Si_2$

J.D. HOOGENDORP and C. ROMERS, 1982. Acta Cryst., B38, 2738-2741.

Orthorhombic,  $P2_12_12_1$ ,  $a = 8.649$ ,  $b = 23.301$ ,  $c = 26.902$  Å (at 293 K),  $a = 8.630$ ,  $b = 23.082$ ,  $c = 26.301$  Å (at 97 K),  $Z = 8$ . Mo radiation,  $R = 0.045$  for 2830 reflexions (at 97 K).

In the two independent molecules in the asymmetric unit (Fig. 1) the ribose rings display a  $C(2')$ -endo conformation; pseudorotation angles  $P = 160.3(2)$  and  $163.2(1)^\circ$  and  $\phi_{max} = 40.5(3)$  and  $41.0(3)^\circ$ , respectively. Both  $O(5')$  atoms are gauche-gauche oriented with respect to  $O(1')$  and  $C(3')$ . The two cytosine bases are anti with respect to the sugar rings:  $\chi_{CN} = 39.9(5)$  and  $55.6(5)^\circ$ . The two independent molecules form dimeric units by means of hydrogen bonds  $N(3A) \dots H-N(4B)$  (2.992(5) Å) and  $N(4A) \dots N(3B)$  (2.967(5) Å). The cytosine carbonyl oxygens serve as acceptors in hydrogen bonds  $O(5'A) \dots H \dots O(2A)$  (2.696(4) Å) and  $O(5'B) \dots H \dots O(2B)$  (2.718(4) Å) connecting neighbouring molecules in the direction [100].

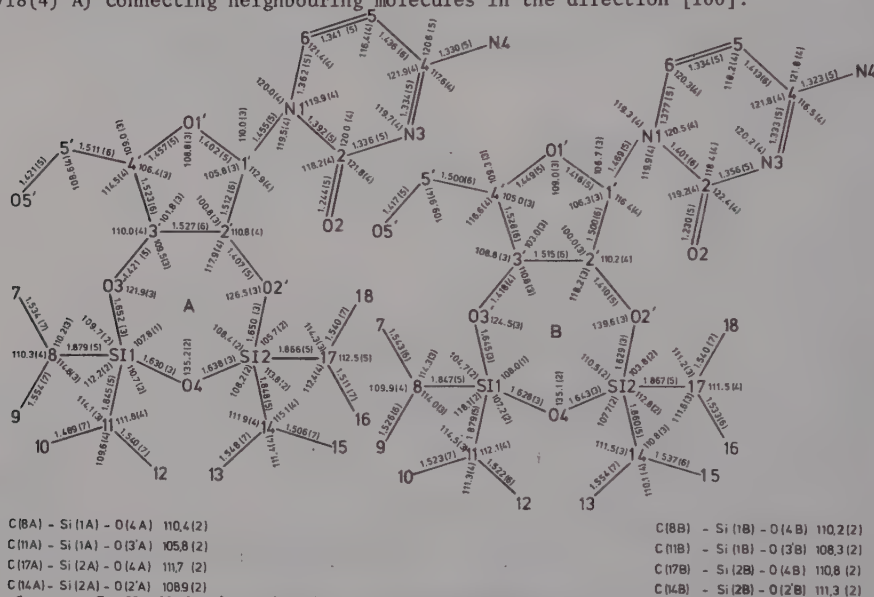
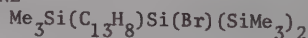
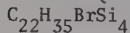


Fig. 1.  $C_{21}H_{39}N_3O_6Si_2$ : bond distances and angles.

BROMO-9-(9-TRIMETHYLSILYLFLUORENYL)BIS(TRIMETHYLSILYL)SILANE



U. SCHUBERT and C. STEIB, 1982. J. Organometal. Chem., 238, C1-C4.

Tetragonal,  $I4_1cd$ ,  $a = 21.010$ ,  $c = 23.995$  Å,  $Z = 16$ . Mo radiation,  $R = 0.068$  for 1164 reflexions.

The overall configuration of the molecule is trans (Si(3) relative to Si(4)) with a  $Si(4)-C(9)-Si(1)-Si(3)$  torsion angle of  $176.3^\circ$ . The geometry about Si(1) is distorted tetrahedral with  $Br-Si(1)-Si(2)$  98.4,  $Br-Si(1)-Si(3)$  103.7,  $Br-Si(1)-C(9)$  108.4,  $Si(2)-Si(1)-Si(3)$  111.7,  $Si(2)-Si(1)-C(9)$  122.1 and  $Si(3)-Si(1)-C(9)$  110.2°. The  $Si(1)-C(9)-Si(4)$  angle is  $117.3^\circ$ .

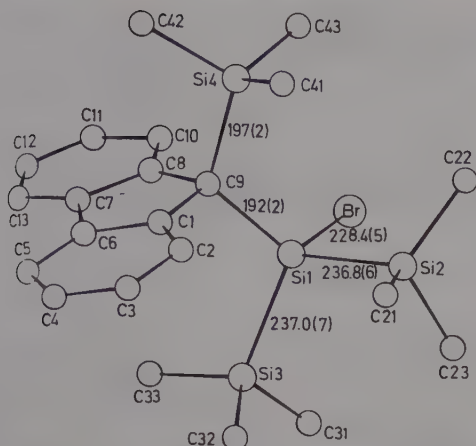


Fig. 1. Molecular structure of  $C_{22}H_{35}BrSi_4$  showing selected bond lengths (pm).

2,2,4,4,6,6-HEXA-*t*-BUTYLCYCLOTRISILOXANE
$$\text{C}_{24}\text{H}_{54}\text{O}_3\text{Si}_3$$

W. CLEGG, 1982. *Acta Cryst.*, B38, 1648-1649.

Rhombohedral,  $R\bar{3}c$ ,  $a = 10.103$ ,  $c = 48.967$  Å,  $Z = 6$ . Mo radiation,  $R = 0.047$  for 1102 reflexions.

The molecule (Fig. 1) has crystallographic 32 ( $D_3$ ) symmetry with a strictly planar  $Si_3O_3$  ring. Bond lengths and angles are normal.

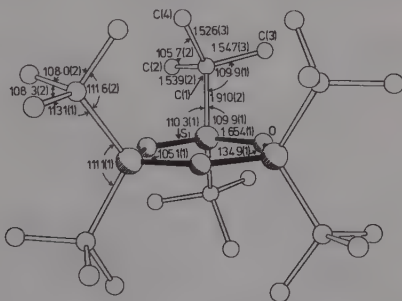


Fig. 1. The 2,2,4,4,6,6-hexa-*t*-butylcyclotrisiloxane molecule.

4-DIMETHYL-2-TRIMETHYLSILOXY-1-TRIMETHYLSILYL-2-(N,N-BIS(TRIMETHYLSILYL)AMINO)-3-(N,N-BIS(TRIMETHYLSILYL)AMINO)(TRIMETHYL(SILOXY)METHYLSILYL)-1,3-DIAZA-2,4-DISILACYCLOBUTANE (ISOMER I)

$$\text{C}_{24}\text{H}_{72}\text{N}_4\text{O}_2\text{Si}_{10} \quad (\text{I})$$

4-DIMETHYL-2-TRIMETHYLSILOXY-1-TRIMETHYLSILYL-2-(N,N-(BIS(TRIMETHYLSILYL))AMINO)-3-(N,N-(BIS(TRIMETHYLSILYL))AMINO)(TRIMETHYL(SILOXY)METHYLSILYL)-1,3-DIAZA-2,4-DISILACYCLOBUTANE (ISOMER II)

$$\text{C}_{24}\text{H}_{72}\text{N}_4\text{O}_2\text{Si}_{10} \quad (\text{II})$$

W. CLEGG, U. KLINGEBIEL and G.M. SHELDRICK, 1982. Z. Naturforsch., 37b, 423-431.

I. Triclinic,  $P\bar{1}$ ,  $a = 12.374$ ,  $b = 13.649$ ,  $c = 13.986$  Å,  $\alpha = 79.05$ ,  $\beta = 87.47$ ,  $\gamma = 78.47^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.045$  for 6530 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 10.713$ ,  $b = 13.297$ ,  $c = 18.236$  Å,  $\alpha = 102.00$ ,  $\beta = 96.65$ ,  $\gamma = 112.22^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.069$  for 4038 reflexions.

The structures of the two diastereoisomers were established as those shown in Fig. 1. In both molecules the central  $\text{Si}_2\text{N}_2$  ring is planar with mean Si-N 1.746(3) Å. The mean exocyclic Si-N and Si-O distances are 1.724(3) and 1.623(3) Å respectively.

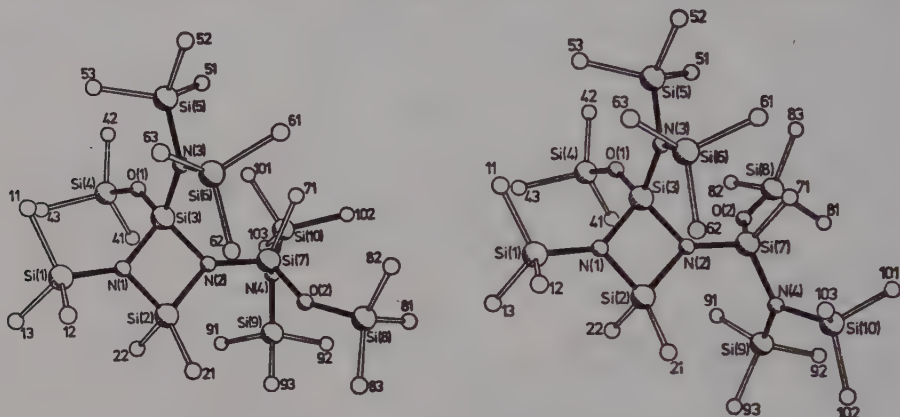


Fig. 1. The structures of  $\text{C}_{24}\text{H}_{72}\text{N}_4\text{O}_2\text{Si}_{10}$ , Isomer I (left) and II (right).

2,2,4,4,6,6,12,12,14,14,16,16,21,21,23,23,25,25,30,30,32,32,34,34-TETRAEICOSIMETHYL-TETRASPIRO[7.1.7.1.7.1.7.1]HEXADECASILAEICOSIOXANE

$\text{C}_{24}\text{H}_{72}\text{O}_{20}\text{Si}_{16}$

V.E. SHKLOVER, Yu.T. STRUCHKOV, A.B. ZACHERNYUK and A.A. ZHDANOV, 1982. Cryst. Struct. Comm., 11, 1771-1775.

Triclinic,  $P\bar{1}$ ,  $a = 8.400$ ,  $b = 13.58$ ,  $c = 13.86$  Å,  $\alpha = 111.55$ ,  $\beta = 104.89$ ,  $\gamma = 90.55^\circ$ ,  $Z = 1$ . Mo radiation,  $R = 0.047$  for 3885 reflexions (at  $-120^\circ\text{C}$ ).

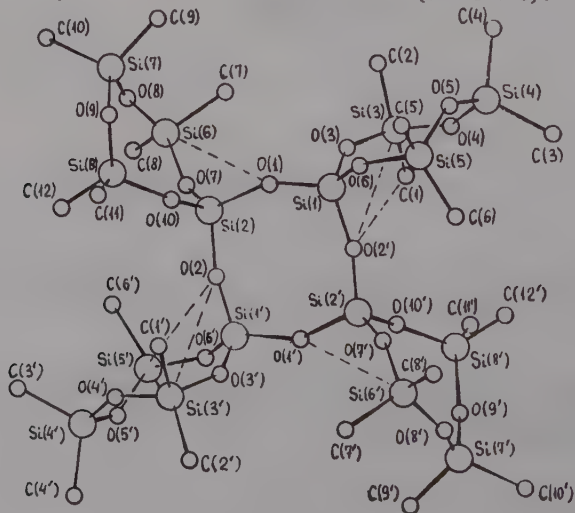


Fig. 1. The  $\text{C}_{24}\text{H}_{72}\text{O}_{20}\text{Si}_{16}$  molecule.

The average Si-O bond length in the spiro-centres (Fig. 1) (1.607(4) Å) is much shorter than the average value for the other Si-O bond lengths (1.629(6) Å). The central centrosymmetric ring is flattened (largest deviation 0.096 Å).

1-(t-BUTYLDIMETHYLSILOXY)-1,2,3,4-TETRAHYDRO-8-METHOXYNAPHTHO(2:3-c)-2-OXO-5-[(6-ACETOXY-3-METHYL-2,4-DIOXACYCLOHEXYL) ACETOXYMETHYL]TETRAHYDROFURAN  
 $C_{29}H_{42}O_{10}Si$

R.W. FRANCK, T.V. JOHN, K. OLEJNICZAK and J.F. BLOUNT, 1982. J. Am. Chem. Soc., 104, 1106-1107.

Monoclinic,  $P2_1$ ,  $a = 13.145$ ,  $b = 7.220$ ,  $c = 17.871$  Å,  $\beta = 108.10^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.044$  for 2096 reflexions (absolute configuration).

A stereoview of the molecule is shown in Fig. 1. No unusual geometrical parameters were noted.

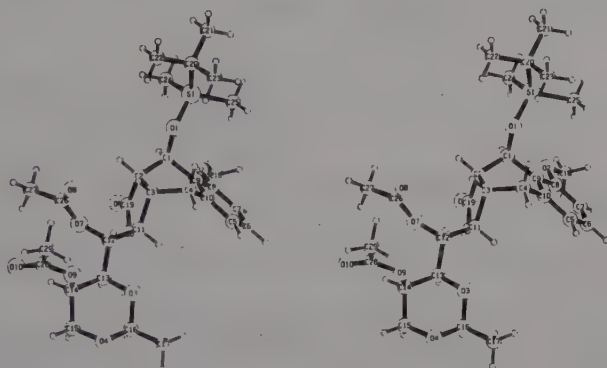


Fig. 1. Stereoview of  $C_{29}H_{42}O_{10}Si$ .

2,2,2,2-TETRACARBONYL-1,1,3,3-TETRAPHENYL-4,5-DIETHYL-1,3,2-DISILAFERRACYCLOPENT-4-ENE  
 $C_{34}H_{30}FeO_4Si_2$  (I)

(3-4)- $\eta^2$ -(6-7)- $\eta^2$ -1,1,1,2,2,2-HEXACARBONYL-5,5-DIPHENYL-3,4,6,7-TETRAMETHYL-1,2,5-DIFERRASILACYCLOHEPTA-3,6-DIENE

$C_{26}H_{22}Fe_2O_6Si$  (II)

F.H. CARRÉ and J.J.E. MOREAU, 1982. Inorg. Chem., 21, 3099-3105.

I. Triclinic,  $P\bar{1}$ ,  $a = 11.748$ ,  $b = 17.246$ ,  $c = 8.602$  Å,  $\alpha = 86.70^\circ$ ,  $\beta = 110.94^\circ$ ,  $\gamma = 107.68^\circ$ ,  $D_m = 1.32$ ,  $Z = 2$ . Mo radiation,  $R = 0.047$  for 1962 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 11.163$ ,  $b = 16.191$ ,  $c = 15.165$  Å,  $\beta = 109.02^\circ$ ,  $D_m = 1.45$ ,  $Z = 4$ . Mo radiation,  $R = 0.038$  for 2105 reflexions.

Both molecules are shown in Fig. 1. In I the Fe atom is coordinated to two Si atoms (Fe-Si 2.405(3) and 2.418(2) Å), and four CO ligands (mean Fe-C 1.782 Å) in a distorted octahedral arrangement (Si-Fe-Si  $81.92(2)^\circ$ ). Molecules of II have approximate  $C_2$  symmetry with an Fe-Fe bond of 2.517(1) Å. Each Fe atom is also coordinated to three terminal CO ligands (mean Fe-C 1.785 Å), a  $\sigma$ -bonded C atom (mean Fe-C 2.003 Å), and to two C atoms via a  $\pi$ -linkage (Fe-C( $\pi$ ) 2.093(5)-2.191(4) Å).

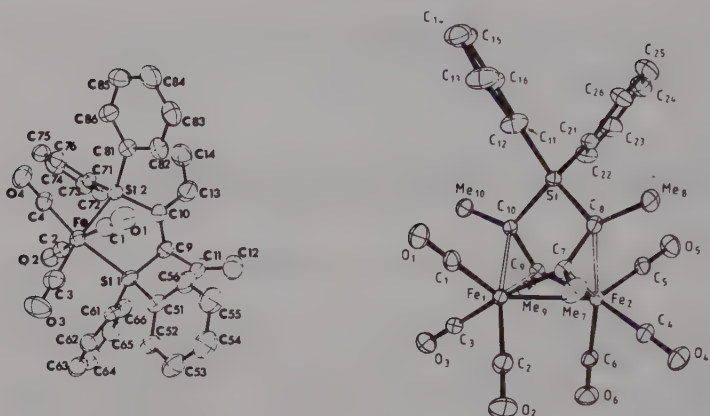


Fig. 1. The  $C_{34}H_{30}FeO_4Si_2$  (left) and  $C_{26}H_{22}Fe_2O_6Si$  (right) molecules.

trans-1,4-DIMETHYL-1,2,4,5-TETRAPHENYL-3,6-BIS(TRIMETHYLSILYL)-1,4-DISILACYCLOHEXA-2,5-DIENE

$C_{36}H_{44}Si_4$

M. ISHIKAWA, H. SUGISAWA, M. KUMADA, T. HIGUCHI, K. MATSUI and K. HIROTSU, 1982. Organometallics, 1, 1473-1477.

Orthorhombic,  $Pbca$ ,  $a = 20.771$ ,  $b = 18.842$ ,  $c = 9.201$  Å,  $Z = 4$ . Mo radiation,  $R = 0.053$  for 2074 reflexions.

The molecule has a crystallographic centre of symmetry (Fig. 1) so the phenyl group on the Si atom in the 1-position of the central ring is trans with respect to the phenyl group in the 4-position. The central ring is in the chair form with the following deviations from the best fit plane: Si -0.013, C(1) 0.017 and C(2) -0.016 Å. Bond distances within the ring are: Si(1)-C(1) 1.893, Si(1)-C(2') 1.878, C(1)-C(2) 1.355 Å, and endocyclic angles are: C(1)-Si(1)-C(2') 113.7, Si(1)-C(1)-C(2) 127.3 and C(1)-C(2)-Si(1') 118.9°. Other bond distances are: Si(1)-C(3) 1.872, C(1)-C(9) 1.510, C(2)-Si(2) 1.904, Si(2)-C(16) 1.850, Si(2)-C(17) 1.853, Si(2)-C(18) 1.848 Å.

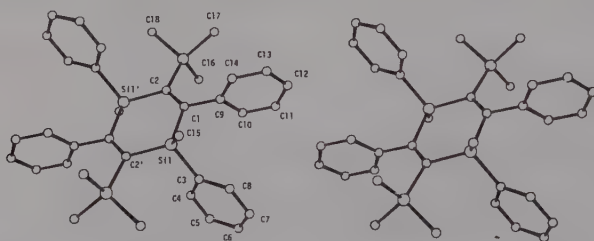


Fig. 1. Stereoscopic view of  $C_{36}H_{44}Si_4$ .

1,1,1,3,3,3-HEXAPHENYL-1,3-DISILAPROPANE

$C_{37}H_{32}Si_2$

$H_2C(SiPh_3)_2$

C. GLIDEWELL and D.C. LILES, 1982. J. Organometal. Chem., 234, 15-25.

Triclinic,  $P\bar{1}$ ,  $a = 9.290$ ,  $b = 12.128$ ,  $c = 16.882$  Å,  $\alpha = 62.08$ ,  $\beta = 106.88$ ,  $\gamma = 117.28^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.11$  for 2134 reflexions.

The structure consists of isolated molecules with their central  $C_3Si...SiC_3$  fragments in staggered arrangements (Fig. 1). The central Si-C distances are 1.83 and 1.87 Å, the Si(1)-C(1)-Si(2) angle  $128.8^\circ$  and the Si(1)...Si(2) distance 3.336 Å. The Si-C(phenyl) distances range from 1.84 to 1.89 Å.

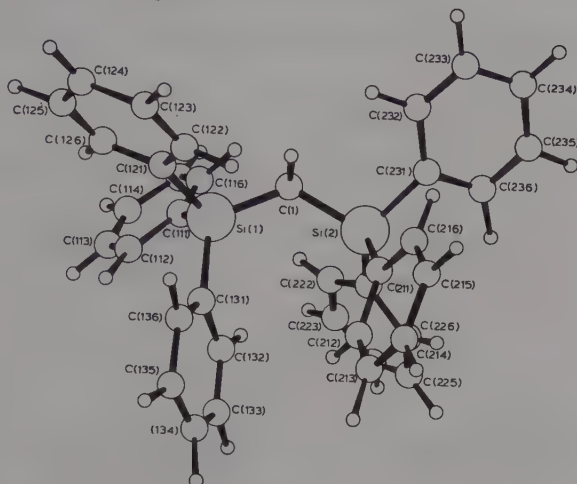


Fig. 1. Perspective view of  $H_2C(SiPh_3)_2$ .

TRIPHENYLISOPROPYLPHOSPHONIUM BIS(TRIMETHYLSILYLMETHYL)FLUOREN-9-YLCHLOROSILANE  
 $C_{42}H_{52}ClPSi_3$

I.V. BORISOVA, N.N. ZEMLYANSKY, V.K. BELSKY, N.D. KOLOSOVA, A.N. SOBOLEV, Y.N. LUZIKOV, Y.A. USTYNYUK and I.P. BELETSKAYA, 1982. J. Chem. Soc. Chem. Commun., 1090-1092.

Monoclinic,  $P2_1$ ,  $a = 10.878$ ,  $b = 10.498$ ,  $c = 10.140$  Å,  $\gamma = 118.24^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.056$  for 866 reflexions.

The compound was clearly shown to be ionic (Fig. 1). In the anion the Si(1)-C(7) bond length (1.83) is significantly shorter than the Si(1)-C(14) and Si(1)-C(15) bond lengths (1.88, 1.89 Å). The Si-Cl bond is 2.107 Å, longer than normal. C(7) is trigonal planar and the fluorene skeleton is normal. The phosphonium cation also has normal geometry.

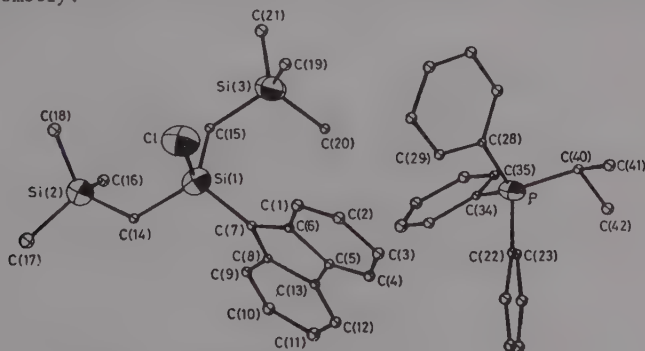


Fig. 1.  $C_{42}H_{52}ClPSi_3$ : the crystal structure.



(1R,3S,5R,6R,7S,8R,10R,11S,14R,15S,18R)-13,14-DIBENZYL-18-[2-(TRIMETHYLSILYL)ETHOXY]-6,7,10-TRIHYDROXY-5,7,16,17-TETRAMETHYL-2,12-DIOXO-13-AZATETRACYCLO-[9.7.0.0<sup>3</sup>,8.0<sup>11,15</sup>]OCTADEC-16-ENE-6,7-ACETONIDE  
 $C_{43}H_{59}NO_6Si$

S.G. PYNE, D.C. SPELLMEYER, S. CHEN and P.L. FUCHS, 1982. J. Am. Chem. Soc., 104, 5728-5740.

Orthorhombic,  $P2_12_12_1$ ,  $a = 8.748$ ,  $b = 18.66$ ,  $c = 25.57 \text{ \AA}$ ,  $D_m = >1.0$ ,  $Z = 4$ . Cu radiation,  $R = 0.078$  for 4467 reflexions.

The structure is shown in Fig. 1. No unusual geometrical features were noted.



Fig. 1. Two stereoviews of  $C_{43}H_{59}NO_6Si$ .

trans-VINYLENEDIPHOSPHONIC ACID HEXAAQUACOBALT(I) DIHYDRATE  
 $C_2H_{16}CoO_{12}P_2 \cdot 2(H_2O)$

E.B. CHUKLANOVA, T.N. POLYNOVA, M.A. PORAI-KOSHITS, M.A. KONSTANTINOVSKAYA and E.I. SINYAVSKAYA, 1982. Koord. Khim., 8, 236-239.

Triclinic,  $P\bar{1}$ ,  $a = 10.370$ ,  $b = 6.603$ ,  $c = 6.512 \text{ \AA}$ ,  $\alpha = 120.21$ ,  $\beta = 90.41$ ,  $\gamma = 104.76^\circ$ ,  $Z = 1$ . Mo radiation,  $R = 0.06$  for 1555 reflexions.

The Co atom, which lies at a crystallographic centre of inversion, is coordinated octahedrally to six water molecules (Co-O 2.066-2.099(5)  $\text{\AA}$ ). In the trans-vinylene-diphosphonic acid moiety the C=C and P-C distances are 1.332(7) and 1.792(6)  $\text{\AA}$  respectively. The P-O distances are 1.500(4), 1.509(4) and 1.575(4)  $\text{\AA}$ . There is extensive hydrogen bonding in the crystal (O...O 2.585(6)-2.847(6)  $\text{\AA}$ ) (Fig. 1).

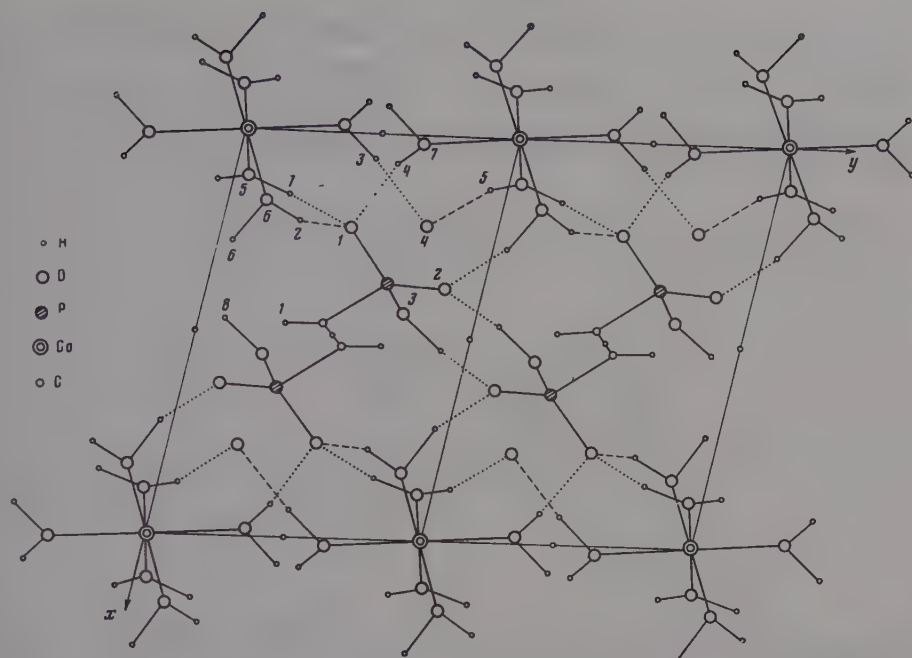


Fig. 1.  $C_2H_{16}CoO_{12}P_2 \cdot 2(H_2O)$ : (001) projection of the crystal structure.

2-(TRICHLOROPHOSHAZO)PERCHLOROPROPANE  
 $C_3Cl_{10}NP$



M.Yu. ANTIPIN, Yu.T. STRUCHKOV, V.M. YURCHENKO and E.S. KOZLOV, 1982. Zh. Strukt. Khim., 23-2, 72-76 [J. Struct. Chem., 23, 227-231].

Monoclinic,  $P2_1/n$ ,  $a = 8.395$ ,  $b = 10.762$ ,  $c = 14.771$  Å,  $\beta = 96.64^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.026$  for 2685 reflexions.

The molecular geometry is shown in Fig. 1. The short  $P=N$  distance of  $1.504(2)$  Å and the large  $P-N-C$  angle of  $144.6(2)^\circ$  are unusual for noncyclic phosphazo compounds.

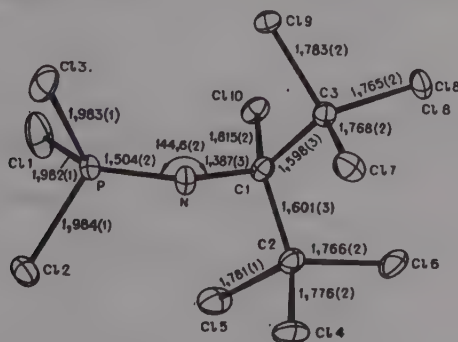


Fig. 1. Bond lengths in  $Cl_3P=NCCl(CCl_3)_2$ .

SPIRO(1,3-DIAZA-2-PHOSPHACYCLOHEXANE-2,2'-4,4,6,6-TETRACHLOROCYCLOTRIPHOSPHAZENE)  
 $C_3H_8Cl_4N_5P_3$

R. ENJALBERT, G. GUERCH, J.-F. LABARRE and J. GALY, 1982. *Z. Krist.*, **160**, 249-258.

Hexagonal,  $P6_122$ .  $a = 9.271$ ,  $c = 26.09$  Å,  $Z = 6$ . Mo radiation,  $R = 0.049$  for 647 reflexions (at 133 K).

The molecule (Fig. 1) has twofold crystallographic symmetry with bond lengths P(1)-N(1) 1.609(5), P(1)-N(3) 1.618(6), P(2)-N(1) 1.553(6), P(2)-N(2) 1.579(2), mean P-Cl 1.990(3), N(3)-C(1) 1.467(9), C(1)-C(2) 1.524(9) Å.

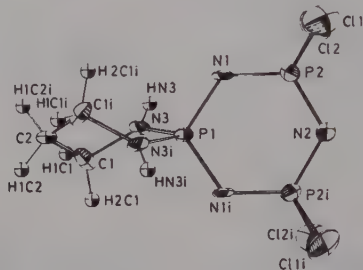


Fig. 1. The  $C_3H_8Cl_4N_5P_3$  molecule.

PENTACHLOROISOPROPYLAMINOCYCLOTRIPHOSPHAZATRIENE  
 $C_3H_8Cl_5N_4P_3$

G.J. BULLEN, 1982. *J. Cryst. Spectrosc. Res.*, **12**, 11-23.

Monoclinic,  $P2_1/n$ ,  $a = 15.059$ ,  $b = 14.815$ ,  $c = 6.501$  Å,  $\beta = 95.03^\circ$ ,  $D_m = 1.699$ ,  $Z = 4$ . Mo radiation,  $R = 0.063$  for 1530 reflexions.

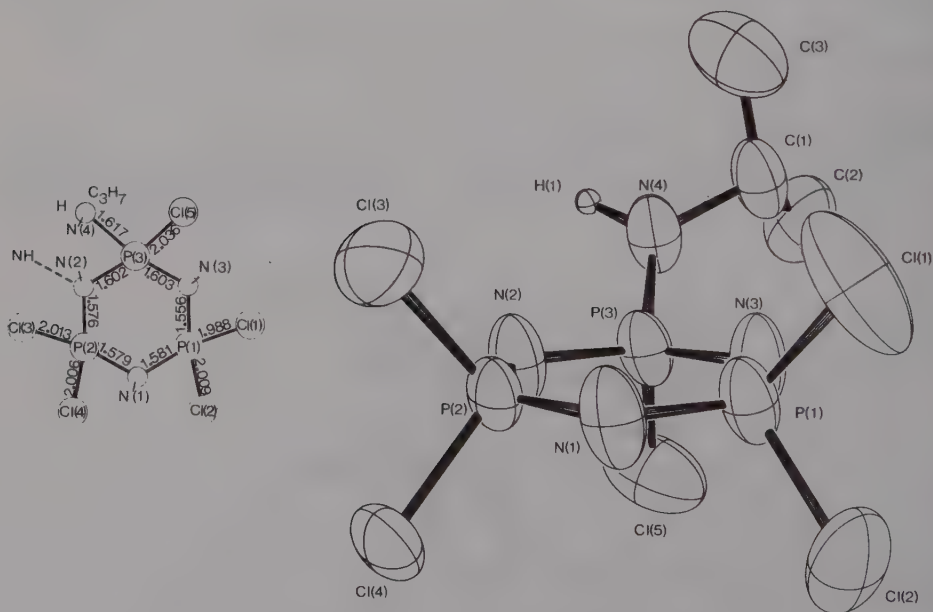
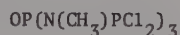
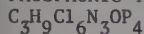


Fig. 1. The structure of  $C_3H_8Cl_5N_4P_3$ .

In the structure (Fig. 1), pairs of molecules mutually related by a centre of symmetry are linked by N-H...N hydrogen bonds. The endocyclic P-N bonds range in length from 1.556 to 1.603 Å depending on the location of the bond relative to the isopropylamino group, and the exocyclic P-N bond is only a little longer (1.617 Å). The nongeminal P-Cl bond is significantly longer (2.036 Å) than the remaining P-Cl bonds (1.988-2.013 Å). The phosphazene ring shows only a very small departure from planarity. The isopropylamino group has a symmetrical conformation, with a Cl-P-N-C torsion angle of 90.5°.

PHOSPHORIC-TRIS (N-DICHLOROPHOSPHINO-N-METHYLAMIDE)



E. FLUCK, S. KLEEMANN, H. HESS and H. RIFFEL, 1982. *Z. Anorg. Chem.*, **486**, 187-192.

Trigonal, R3c,  $a = 15.310$ ,  $c = 22.622$  Å,  $Z = 2$ . Mo radiation,  $R = 0.020$  for 1170 reflexions (at 173 K).

The structure contains two independent molecules (Fig. 1) each with  $C_3$  symmetry. The central P atom is tetrahedrally coordinated with P-O 1.461(8) and P-N 1.674(4) Å. The remaining P atoms are trigonal-pyramidal with P-N 1.694(3) and P-Cl 2.066(2) Å.

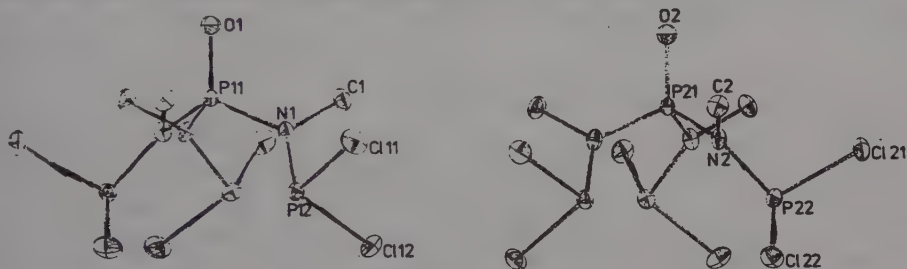
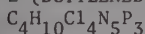


Fig. 1. The two independent molecules of  $\text{C}_5\text{H}_9\text{Cl}_6\text{N}_3\text{OP}_4$ .

2-(BUTYLENEDIAMINE-N,N')-4,4,6,6-TETRACHLORO-CYCLOTRIPOSPHAZENE



G. GUERCH, J.-F. LABARRE, R. ROQUES and F. SOURNIES, 1982. *J. Mol. Struct.*, **96**, 113-120.

Monoclinic,  $P2_1/c$ ,  $a = 10.463$ ,  $b = 17.174$ ,  $c = 16.239$  Å,  $\beta = 102.53^\circ$ ,  $Z = 8$ .  $R = 0.10$  for three-dimensional data.

The analysis establishes the spiro geometry shown in Fig. 1 for the two independent molecules in the asymmetric unit. The analysis is of low accuracy because of crystal decomposition. P-N bond lengths are in the range 1.55-1.62(5) Å.

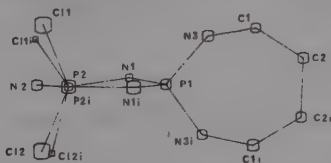


Fig. 1. A side-on view of  $\text{C}_4\text{H}_{10}\text{Cl}_4\text{N}_5\text{P}_3$ .

1,1,5,5-TETRAMETHYLBICYCLO[3.3.0]-1,5-DIPHOSPHA-3,7-DITHIA-2,4,6,8-TETRAZENE  
 $C_4H_{12}N_4P_2S_2$   $(CH_3)_2P(NSN)_2P(CH_3)_2$

N. BURFORD, T. CHIVERS, P.W. CODDING and R.T. OAKLEY, 1982. *Inorg. Chem.*, **21**, 982-986.

Orthorhombic,  $Pnma$ ,  $a = 11.081$ ,  $b = 8.216$ ,  $c = 11.837$  Å,  $Z = 4$ . Mo radiation,  $R = 0.033$  for 692 reflexions (at  $-100^\circ C$ ).

The structure (Fig. 1) consists of a folded eight-membered ring (butterfly) with a cross-ring S-S contact of  $2.551(2)$  Å. The angle between the two intersecting planes of the eight-membered ring is  $114.9(2)^\circ$ . The mean endocyclic P-N and S-N bond lengths are  $1.636(3)$  and  $1.595(3)$  Å.

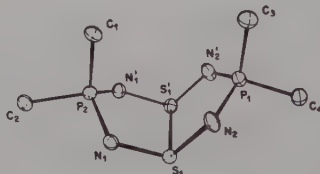


Fig. 1. The  $(CH_3)_2P(NSN)_2P(CH_3)_2$  molecule.

1-METHYLPYRROL-2-YLHEPTAFLUOROCYCLOTETRAPHOSPHAZENE  
 $C_5H_6F_7N_5P_4$

R.D. SHARMA, S.J. RETTIG, N.L. PADDOCK and J. TROTTER, 1982. *Canad. J. Chem.*, **60**, 535-541.

Monoclinic,  $P2_1/n$ ,  $a = 17.300$ ,  $b = 5.028$ ,  $c = 16.902$  Å,  $\beta = 108.904^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.038$  for 1167 reflexions.

The  $N_4P_4$  ring (Fig. 1) is nearly planar and the P-N bond lengths corrected for libration range from  $1.524(5)$  to  $1.562(5)$  Å with a mean value  $1.541(17)$  Å. Mean ring angles are  $122.9(20)$  at P and  $146.4(23)^\circ$  at N. The P1-C1 bond ( $1.756(5)$  Å) is significantly shorter than in typical P-phenyl derivatives and the P1-F1 bond is long ( $1.561(4)$  Å), consistent with conjugative electron release from the pyrrol group, involving  $p_\pi-d_\pi$  bonding.

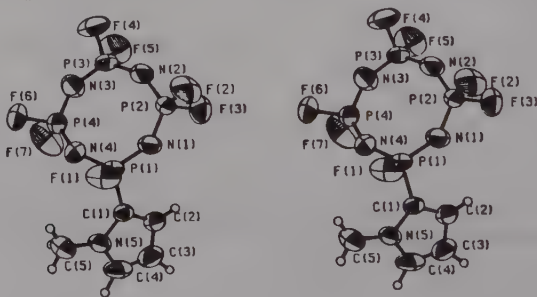


Fig. 1. A stereoview of  $C_5H_6F_7N_5P_4$ .

2-THIOXO-2-METHYL-1,3,6,2-TRIOXAPHOSPHOCANE  
 $C_5H_{11}O_3PS$

A. GRAND, 1982. *Cryst. Struct. Comm.*, **11**, 569-574.

Monoclinic,  $P2_1/m$ ,  $a = 7.570$ ,  $b = 8.063$ ,  $c = 8.204$  Å,  $\beta = 120.83^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.067$  for 846 reflexions.

The eight-membered ring has a crown conformation (Fig. 1) with a crystallographic mirror plane passing through P, S, C(6) and O(5). The P atom has distorted tetrahedral coordination, with P-S 1.914(3), P-O(2) 1.584(3), P-C(6) 1.773(7) Å, O-P-O 105.8(2), O-P-S 115.9(1), C-P-S 115.6(3), P-O-C 123.4(3)°.

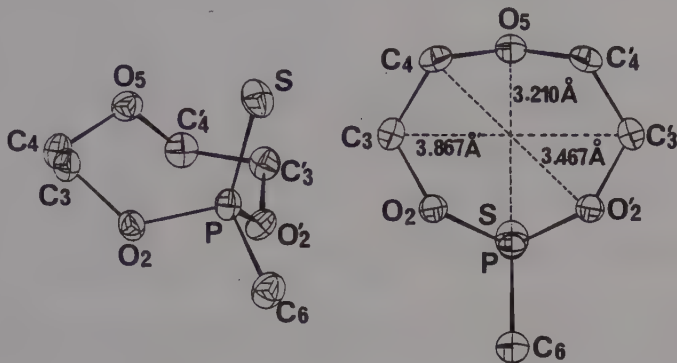


Fig. 1. Views of  $C_5H_{11}O_3PS$ .

2-AMMONIO-4-METHYLPHOSPHINICO-BUTYRATE  
 $C_5H_{12}NO_4P$

E.F. PAULUS and S. GRABLEY, 1982. *Z. Krist.*, **160**, 63-68.

Monoclinic,  $P2_1$ ,  $a = 5.527$ ,  $b = 8.143$ ,  $c = 9.275$  Å,  $\beta = 98.04^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.028$  for 1987 reflexions.

In the crystalline state, the  $\alpha$ -carbon and the phosphorous atoms are chiral, with S and R configuration respectively (Fig. 1). Main bond lengths are P-O(1) 1.532(1), P-O(2) 1.499(1), P-C(1) 1.810(7), P-C(5) 1.767(2) Å. There is a short (2.427 Å) intermolecular hydrogen bond between O(1)-H and O(3), as well as N-H...O hydrogen bonds 2.750 and 2.864 Å.

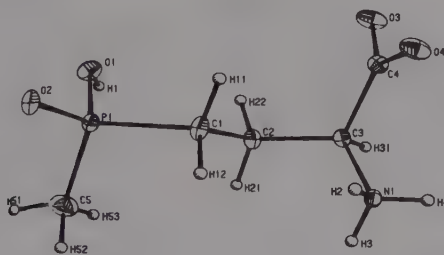


Fig. 1. The  $C_5H_{12}NO_4P$  molecule.

Racemic (3-AMMONIO-3-CARBOXY-PROPYL)METHYLPHOSPHINATE HYDRATE  
 $C_5H_{12}NO_4P \cdot H_2O$

E.F. PAULUS and S. GRABLEY, 1982. *Z. Krist.*, **160**, 39-44.

Monoclinic,  $P2_1/n$ ,  $a = 6.368$ ,  $b = 18.608$ ,  $c = 8.442$  Å,  $\beta = 112.77^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.042$  for 1930 reflexions.



The compound is the betain ammoniumphosphinate (Fig. 1), with P-O(1) 1.534(1), P-O(2) 1.505(1), P-C(1) 1.803(2), P-C5 1.790(2) Å. Molecules are linked into centrosymmetric dimers via O(4)-H...O(1) hydrogen bonds (2.576 Å), which are in turn interlinked by a network of N-H...O (2.662-2.828 Å) and O-H...O (2.867 and 2.752 Å) hydrogen bonds.

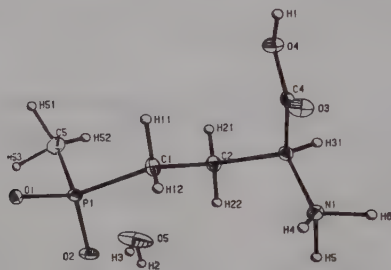


Fig. 1. The racemic  $C_5H_{12}NO_4P \cdot H_2O$  structure.

4,6,9-TRIMETHYL-1,3,4,6,7,9-HEXAAZA-5-PHOSPHATRICYCLO[3.3.1.1<sup>3,7</sup>]DECANE  
 $C_6H_{15}N_6P$

J. JAUD, M. BENHAMMOU, J.P. MAJORAL and J. NAVECH, 1982. *Z. Krist.*, **160**, 69-79.

Monoclinic,  $P2_1/n$ ,  $a = 8.742$ ,  $b = 11.342$ ,  $c = 10.209$  Å,  $\beta = 90.28^\circ$ ,  $D_m = 1.3$ ,  $Z = 4$ . Mo radiation,  $R = 0.052$  for 2615 reflexions.

In the molecule (Fig. 1) which has approximate threefold symmetry the P-N distances are 1.700, 1.707 and 1.715(3) Å, and it is suggested that the P atom is involved in bonding to adjacent atoms largely via its 3p orbitals and that the lone pair is largely in the 3s orbital.

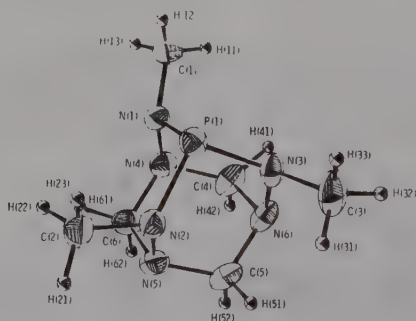


Fig. 1. A view of  $C_6H_{15}N_6P$ .

BIS(TRIMETHYLPHOSPHINE)IMINIUM BROMIDE MONOHYDRATE  
 $C_6H_{18}BrNP_2 \cdot H_2O$



D.W.H. RANKIN, M.D. WALKINSHAW and H. SCHMIDBAUR, 1982. *J. Chem. Soc. Dalton*, 2317-2320.

Triclinic,  $P\bar{1}$ ,  $a = 9.685$ ,  $b = 11.449$ ,  $c = 18.173$  Å,  $\alpha = 90.55^\circ$ ,  $\beta = 91.50^\circ$ ,  $\gamma = 108.92^\circ$ ,  $Z = 6$ . Cu radiation,  $R = 0.059$  for 4103 reflexions.

There are three crystallographically independent sets of cations, anions and water molecules in the asymmetric unit. The cations have similar structures but

differ in conformation (Fig. 1). Anions and water molecules are hydrogen bonded to form  $\text{Br}_6(\text{H}_2\text{O})_6^{6-}$  units. Dimensions in the cation are: P-C 1.779 (mean), P=N 1.582 Å, C-P-C 106.7, P-N-P 140.1, 135.9, 135.1°. The eighteen methyl groups are staggered with respect to their C-P bonds. There are no short intermolecular van der Waals contacts.

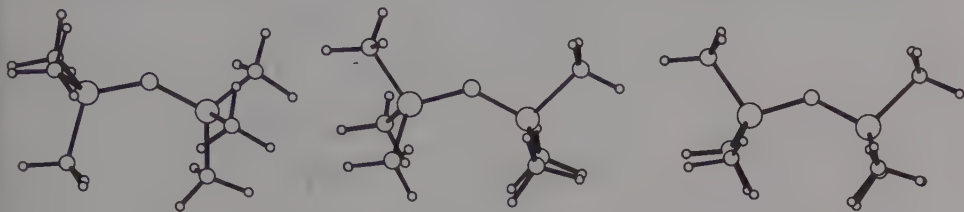


Fig. 1.  $\text{C}_6\text{H}_{18}\text{BrNP}_2 \cdot 2\text{H}_2\text{O}$ : the three crystallographically distinct cations.

DITHIO-closo-TETRAPHOSPHORUS HEXAKIS(METHYLIMIDE)

$\text{C}_6\text{H}_{18}\text{N}_6\text{P}_4\text{S}_2$  (I)

$\text{S}_2\text{P}_4(\text{NCH}_3)_6$

TRITHIO-closo-TETRAPHOSPHORUS HEXAKIS(METHYLIMIDE)

$\text{C}_6\text{H}_{18}\text{N}_6\text{P}_4\text{S}_3$  (II)

$\text{S}_3\text{P}_4(\text{NCH}_3)_6$

F.A. COTTON, J.G. RIESS, C.E. RICE and B.R. STULTS, 1982. *Inorg. Chem.*, **21**, 3123-3126.

I. Monoclinic,  $\text{P}2_1/\text{n}$ ,  $a = 12.582$ ,  $b = 13.110$ ,  $c = 9.747$  Å,  $\beta = 95.00^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.053$  for 2213 reflexions.

II. Orthorhombic,  $\text{Pbca}$ ,  $a = 9.839$ ,  $b = 13.383$ ,  $c = 25.839$  Å,  $Z = 8$ . Mo radiation,  $R = 0.074$  for 1379 reflexions.

The structural series  $\text{S}_n\text{P}_4(\text{NMe})_6$  ( $n = 0-4$ ) is completed by those reported here ( $n = 2, 3$ ; Fig. 1). Data for all five compounds (1) yield the following mean parameters: P-NP 1.70(1), P-NPS 1.73(2), SP-NP 1.65(2) and SP-NPS 1.68(2) Å. Nitrogen atoms are planar in each case, the P-N bonds having bond orders of  $\sim 1.2-1.3$ .

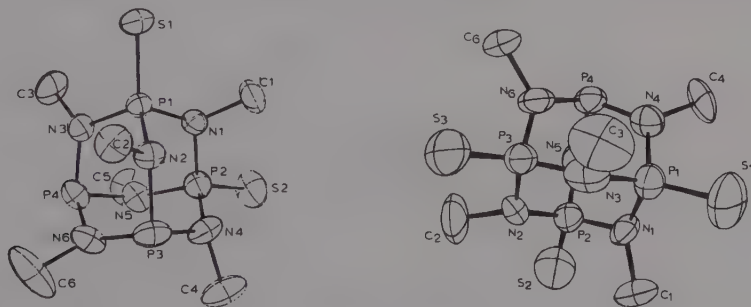


Fig. 1. The  $\text{S}_2\text{P}_4(\text{NCH}_3)_4$  (left) and  $\text{S}_3\text{P}_4(\text{NCH}_3)_4$  (right) molecules.

1. *Structure Reports*, **44B**, 603.

TETRAMETHYLAMMONIUM 2-OXO-2-THIO-1,3,2-OXAZAPHOSPHORINANE

$\text{C}_7\text{H}_{19}\text{N}_2\text{O}_2\text{PS}$

$\text{Et}_4\text{N}^+$ ,  $\text{C}_3\text{H}_7\text{NO}_2\text{PS}^-$

M. MIKOŁAJCZYK, J. OMELANCZUK, W.S. ABDUKAKHAROV, A. MILLER, M.W. WIECZOREK and J. KAROLAK-WOJCIECHOWSKA, 1982. *Tetrahedron*, **38**, 2183-2188.

Orthorhombic,  $P2_12_12_1$ ,  $a = 12.303$ ,  $b = 9.041$ ,  $c = 10.419$  Å,  $D_m = 1.303$ ,  $Z = 4$ . Cu radiation,  $R = 0.0734$  for 925 reflexions.

The anion (Fig. 1) is in chair form with S axial and O(1) equatorial. Main bond lengths are P-S 1.962(3), P-O(1) 1.475(6), P-N(1) 1.675(5), P-O(2) 1.600(5) Å.

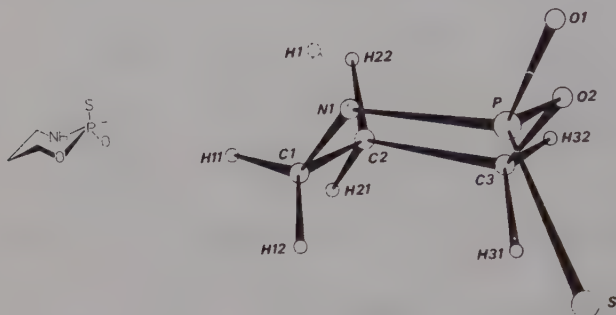


Fig. 1. The  $C_3H_7NO_2PS^-$  anion.

O,O'-DIMETHYL O''-(2,5-DICHLORO-4-IODOPHENYL)PHOSPHOROTHIOATE

$C_8H_8Cl_2IO_3PS$

$(OCH_3)_2P(S)OC_6H_2Cl_2I$

R.G. BAUGHMAN and P.-J. YU, 1982. J. Agric. Food. Chem., **30**, 293-295.

Monoclinic,  $P2_1/c$ ,  $a = 11.999$ ,  $b = 8.263$ ,  $c = 14.032$  Å,  $\beta = 98.89^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.052$  for 1479 reflexions.

The non-hydrogen atoms constituting the phenoxy group are essentially planar (Fig. 1). Bond lengths and angles of note are: C(4)-I 2.09, C(2)-C(1) 1.74, C(5)-C(1) 1.72, C(1)-O(1) 1.36, P-O(1) 1.628, P-O(2) 1.548, P-O(3) 1.561, P-S 1.901, O(2)-C(7) 1.44, O(3)-C(8) 1.46 Å, C(1)-O(1)-P 123.0°. In addition, the P...H(1) and P...H(2) distances (considered important as a possible measure of toxicity/activity of organophosphate insecticides) are respectively 5.589 and 3.133 Å.

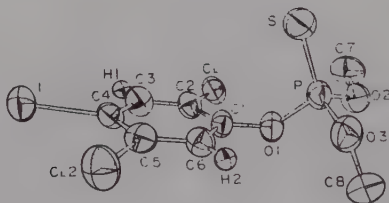


Fig. 1. Molecular structure of  $C_8H_8Cl_2IO_3PS$ .

DIMETHYLPHENYLPHOSPHINE OXIDE

$C_8H_{11}OP$

$C_6H_5P(O)(CH_3)_2$

V.V. TKACHEV, L.O. ATOVMYAN, L.V. GONCHAROVA, A.A. SHVETS and O.A. OSIPOV, 1982. Zh. Strukt. Khim., **23**-4, 168-170 [J. Struct. Chem., **23**, 640-642].

Orthorhombic,  $Pbca$ ,  $a = 21.350$ ,  $b = 7.179$ ,  $c = 11.244$  Å,  $Z = 8$ . Cu radiation,  $R = 0.043$  for 719 reflexions.

The geometry of the molecule is shown in Fig. 1.

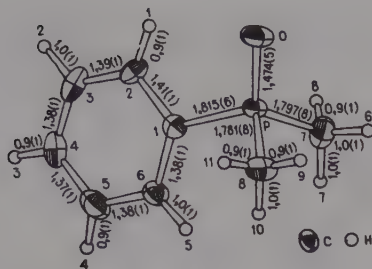
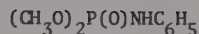
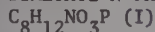
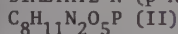


Fig. 1. The  $C_8H_{11}OP$  molecule.

DIMETHYL N-PHENYLPHOSPHORAMIDATE



DIMETHYL N-(p-NITROPHENYL)PHOSPHORAMIDATE



M.P. DU PLESSIS, T.A. MODRO and L.R. NASSIMBENI, 1982. *Acta Cryst.*, B38, 1504-1507.

I. Monoclinic,  $P2_1/n$ ,  $a = 16.929$ ,  $b = 7.706$ ,  $c = 15.883$  Å,  $\beta = 90.24^\circ$ ,  $D_m = 1.28$ ,  $Z = 8$ . Mo radiation,  $R = 0.064$  for 2148 reflexions.

II. Orthorhombic,  $Pbcn$ ,  $a = 24.74$ ,  $b = 7.421$ ,  $c = 12.327$  Å,  $D_m = 1.42$ ,  $Z = 8$ . Mo radiation,  $R = 0.075$  for 719 reflexions.

I has two independent, centrosymmetric, hydrogen-bonded dimers in the unit cell (Fig. 1) with  $N(1) \dots O(1)$  2.88(1) and 2.86(1) Å. II contains monomers linked by  $N(1) \dots O(1)$  hydrogen bonds of length 2.83(5) Å. Both compounds crystallise in the syncoplanar orientation of the phosphoryl and N-H groups. Both compounds exhibit typical geometry e.g. P-N 1.624(4), 1.605(4) in I, 1.643(7) in II, N-C(aromatic) 1.427(6), 1.414(6) in I, 1.405(9) in II, P=O 1.458(3), 1.452(4) in I, 1.462(5) Å in II. The C(3)-C(4)-C(5) angle is  $124.7(10)^\circ$  in II.

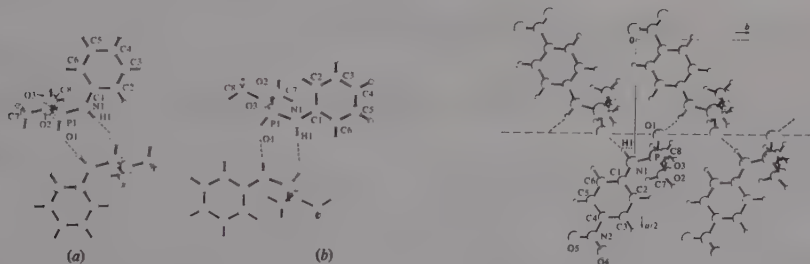
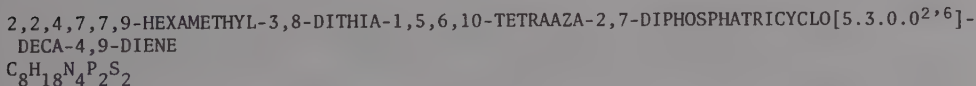


Fig. 1. The two independent dimers of  $C_8H_{12}NO_3P$  (left) and the  $C_8H_{11}N_2O_5P$  molecule showing hydrogen bonding (right).



A. SCHMIDPETER, J. GROSS, E. SCHRENK and W.S. SHELDRIK, 1982. *Phosphorus and Sulfur*, 14, 49-61.

Triclinic,  $P\bar{1}$ ,  $a = 7.251$ ,  $b = 8.849$ ,  $c = 6.169$  Å,  $\alpha = 96.57$ ,  $\beta = 98.16$ ,  $\gamma = 111.78^\circ$ ,  $Z = 1$ . Mo radiation,  $R = 0.072$  for 1498 reflexions.

The molecule (Fig. 1) is centrosymmetric and the three rings lie in one plane. The trigonal-bipyramidal coordination of phosphorus is "anti-Berry" distorted and the axial P-S bond is long (2.31 Å). The P-N distances are 1.680 and 1.794(3) Å, with P-C 1.802 and 1.807(5) Å.

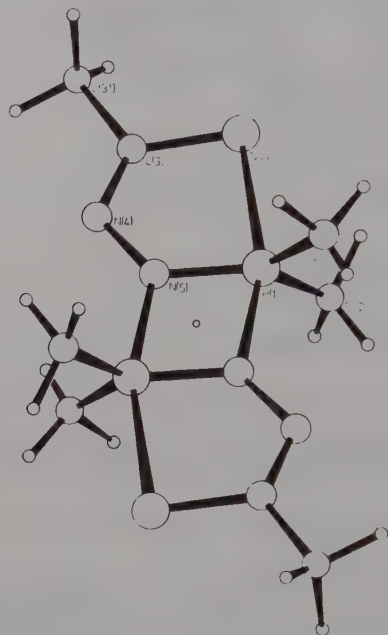


Fig. 1. A view of  $C_8H_{18}N_4P_2S_2$ .

TETRAETHYLAMMONIUM TRICHLOROPHOSPHORUS(III) CHLORIDE

$C_8H_{20}Cl_4NP$  (I)

$(C_2H_5)_4N^+, PCl_4^-$

TRIPHENYLPHOSPHORANYLIDENEMETHYLTRIPHENYLPHOSPHONIUM TRICYANOPHOSPHORUS(III) CHLORIDE

$C_{40}H_{31}ClN_3P_3, C_2H_3N$  (II)

$((C_6H_5)_3P)_2CH^+, P(CN)_3Cl^-, CH_3CN$

K.B. DILLON, A.W.G. PLATT, A. SCHMIDPETER, F. ZWASCHKA and W.S. SHELDRIK, 1982.  
Z. Anorg. Chem., 488, 7-26.

I. Monoclinic,  $P2_1/n$ ,  $a = 9.006$ ,  $b = 9.129$ ,  $c = 8.926$  Å,  $\beta = 91.68^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.085$  for 939 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 12.457$ ,  $b = 14.669$ ,  $c = 11.927$  Å,  $\alpha = 95.65^\circ$ ,  $\beta = 113.31^\circ$ ,  $\gamma = 104.22^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.054$  for 5390 reflexions.

In structure I (Fig. 1) there is an overall  $C_2$  symmetry but the anions and cations are both disordered so that neither individually has  $C_2$  symmetry. The  $PCl_4^-$  anion adopts a pseudo-trigonal-bipyramidal coordination in which one axial P-Cl bond (2.850(4) Å) is significantly longer than the other (2.118(4) Å); the mean P-Cl(eq.) is 2.046(4) Å. The Cl(1)PCl(2) angle is  $171.4(2)^\circ$ . In II (Fig. 1) the  $P(CN)_3Cl^-$  anion also adopts the pseudo-trigonal-bipyramidal coordination: P-Cl 2.625(1), P-C(axial) 1.915(5), P-C(eq.) 1.783(5) Å, Cl-P-C  $168.2(1)^\circ$ . The bond lengths and angles in the cation agree with expected values.

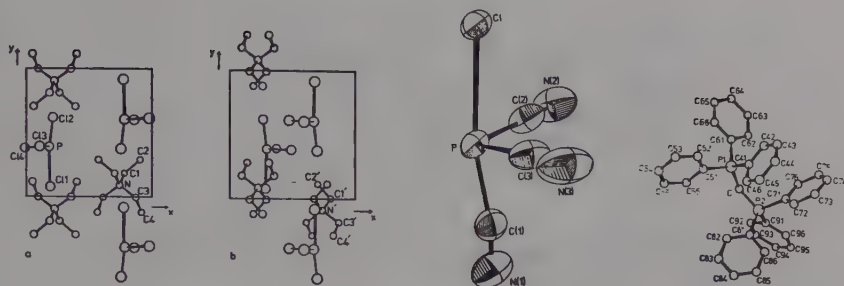


Fig. 1. The disordered  $(C_2H_5)_4P^+ \cdot PCl_4^-$  structure (left), and the  $P(CN)_3Cl^-$  anion and  $((C_6H_5)_3P)_2CH^+$  cation (right).

N-BENZOYLDIMETHYLPHOSPHORAMIDATE  
 $C_9H_{12}NO_4P$

V. MIZRAHI and T.A. MODRO, 1982. Cryst. Struct. Comm., 11, 627-631.

Monoclinic,  $P2_1/n$ ,  $a = 8.080$ ,  $b = 22.16$ ,  $c = 6.347$  Å,  $\beta = 90.78^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.058$  for 1410 reflexions.

Main bond lengths in the molecule (Fig. 1) are P-O(1) 1.461(4), P-O(2) 1.557(4), P-O(3) 1.540(4), P-N(1) 1.667(5) Å. The phenyl ring is twisted with respect to the C(O)NH plane by  $18.5^\circ$ . Molecules are linked in pairs by N-H...O=P hydrogen bonds.

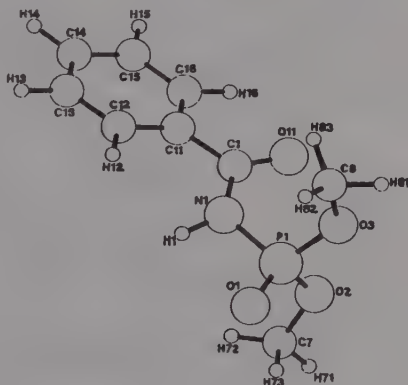


Fig. 1. A view of  $C_9H_{12}NO_4P$ .

TRIS(2-CYANOETHYL)PHOSPHINE OXIDE  
 $C_9H_{12}N_3OP$

B.M. FOXMAN, C.H. KIM and H. MAZUREK, 1982. Acta Cryst., B38, 1622-1624.

Rhombohedral,  $R3c$ ,  $a = 13.501$ ,  $c = 10.177$  Å,  $D_m = 1.30$ ,  $Z = 6$ . Cu radiation,  $R = 0.033$  for 615 reflexions.

The molecule (Fig. 1) has crystallographic threefold symmetry along the P-O bond. The P=O bond length is similar to that found in other phosphine oxides containing alkyl groups. For other work, see (1).



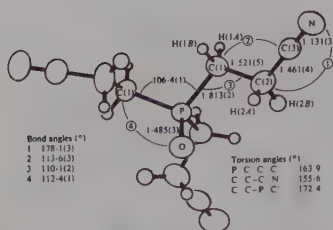
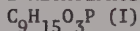


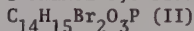
Fig. 1. Bond lengths and angles in tris(2-cyanoethyl)phosphine oxide.

1. Structure Reports, 48B, 827.

1-METHYLPHOSPHONANE-3,8-DIONE 1-OXIDE



1-PHENYL-5,6-DIBROMOPHOSPHONANE-3,8-DIONE 1-OXIDE



L.D. QUIN, E.D. MIDDLEMAS, N.S. RAO, R.W. MILLER and A.T. McPHAIL, 1982. J. Am. Chem. Soc., 104, 1893-1900.

I. Monoclinic,  $P2_1$ ,  $a = 10.014$ ,  $b = 9.639$ ,  $c = 5.229$  Å,  $\beta = 90.95^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.043$  for 893 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 12.793$ ,  $b = 10.329$ ,  $c = 12.922$  Å,  $\beta = 113.68^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.073$  for 1575 reflexions.

The two molecules are shown in Fig. 1. The 9-membered rings in both I and II exist in twist chair-chair forms in the solid state. In II there is a nonequivalence of comparable ring carbons, implying the existence of a strongly biased equilibrium or a high barrier to ring inversion.

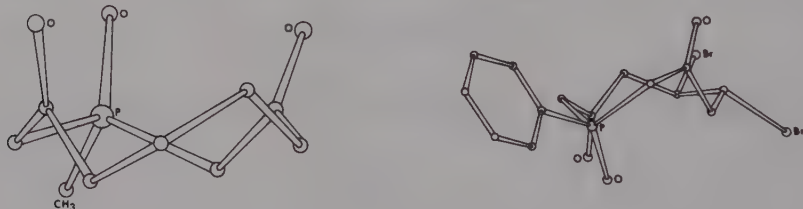
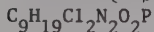


Fig. 1. The structures of  $C_9H_{15}O_3P$  (left) and  $C_{14}H_{15}Br_2O_3P$  (right).

2-OXO-BIS(2-8-CHLOROETHYLAMINO)-4,6-DIMETHYL-1,3,2-OXAZAPHOSPHORINANE



L.E. CARPENTER, D. POWELL, R.A. JACOBSON and J.G. VERKADE, 1982. Phosphorus and Sulfur, 12, 287-293.

Monoclinic,  $P2_1/n$ ,  $a = 11.11$ ,  $b = 7.808$ ,  $c = 16.88$  Å,  $\beta = 105.3^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.106$  for 1359 reflexions.

The six-membered ring has a chair conformation, with an axial mustard group and equatorial oxygen (Fig. 1). Neither pendant  $ClCH_2CH_2$  group is under the ring. Main bond lengths are P-O1 1.475(6), P-O2 1.573(7), P-N1 1.654(8), P-N2 1.621(9) Å.

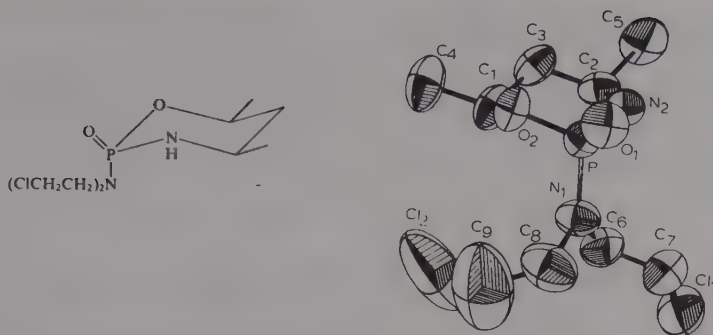


Fig. 1. The  $C_9H_{19}Cl_2N_2O_2P$  molecule.

1,2,3-TRIMETHYL-3',3',5',5'-TETRAKIS(TRIFLUOROMETHYL)SPIRO(1,3,2-DIAZAPHOSPHETIDINE-2,2'-1',4',2'-DIOXAPHOSPHOLANE)-4-ONE

$C_{10}H_9F_{12}N_2O_3P$

N. WEFERLING, R. SCHMUTZLER and W.S. SHELDRIK, 1982. Justus Liebigs Ann. Chem., 167-181.

Triclinic,  $P\bar{1}$ ,  $a = 7.380$ ,  $b = 15.993$ ,  $c = 7.400$  Å,  $\alpha = 99.18$ ,  $\beta = 107.28$ ,  $\gamma = 97.15^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.045$  for 2551 reflexions.

The P atom is trigonal-bipyramidally coordinated (Fig. 1) with P-O(2) 1.718(1), N(4) 1.664(2), N(1) 1.789(2), C(3) 1.784(3), C(5) 1.903(3) Å. The four- and five-membered rings involving P are nearly planar; deviations are: (four-membered ring) P 0.018, N(1) -0.022, C(11) 0.027, N(4) -0.023 Å; (five-membered ring) P 0.031, O(2) -0.034, C(2) 0.016, O(5) 0.016, C(5) -0.029 Å.

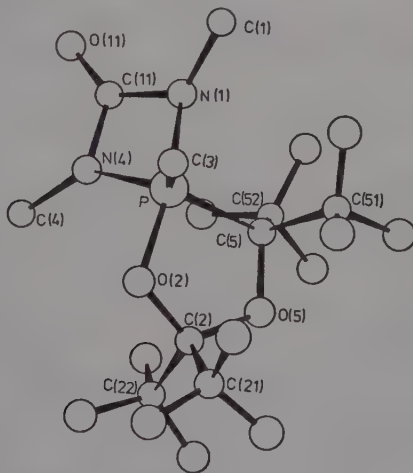


Fig. 1. Perspective view of  $C_{10}H_9F_{12}N_2O_3P$ .

PHOSPHINDOLIN-3-ONE

$C_{10}H_{11}O_3P$

B.R. STULTS, F.L. MAY and T.M. BALTHAZOR, 1982. Cryst. Struct. Comm., 11, 1179-1183.

Monoclinic,  $P2_1$ ,  $a = 5.220$ ,  $b = 16.839$ ,  $c = 5.831$  Å,  $\beta = 96.85^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.041$  for 1160 reflexions.

The molecule (Fig. 1) exists in the keto form. The C(2)-O(3) distance (1.210(6) Å) and the C(1)-C(2) distance (1.521(6) Å) are as expected. The bicyclic portion of the molecule is essentially planar (the nine atoms of the bicyclic ring are within 0.03 Å of the mean plane). The P-O(1)-C(3)-C(4) group is nearly perpendicular to the bicyclic plane (dihedral angle  $102.2^\circ$ ).

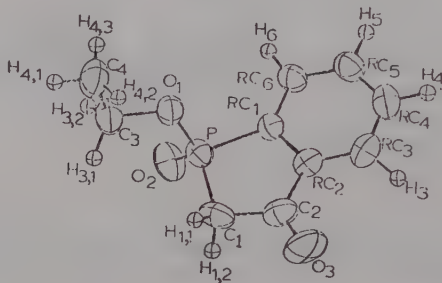


Fig. 1. The  $C_{10}H_{11}O_3P$  molecule.

5-METHYL-2-METHOXY-2-OXO-3-PHENYL-1,3,2-OXAZAPHOSPHOLAN  
 $C_{10}H_{14}NO_3P$  (I)

2,5-DIMETHYL-2-OXO-3-PHENYL-1,3,2-OXAZAPHOSPHOLAN  
 $C_{10}H_{14}NO_2P$  (II)

A.S. BATANOV, Yu.T. STRUCHKOV, M.A. PUDOVIK, L.K. KIBARDINA and A.N. PUDOVIK, 1982. Kristallografiya, 27, 262-266 [Sov. Phys. Crystallogr., 27, 160-163].

I. Monoclinic,  $C2/c$ ,  $a = 14.826$ ,  $b = 11.711$ ,  $c = 12.853$  Å,  $\beta = 98.67^\circ$ ,  $Z = 8$ . Cu radiation,  $R = 0.038$  for 1291 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 18.918$ ,  $b = 8.15$ ,  $c = 6.891$  Å,  $\beta = 100.37^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.114$  for 926 reflexions.

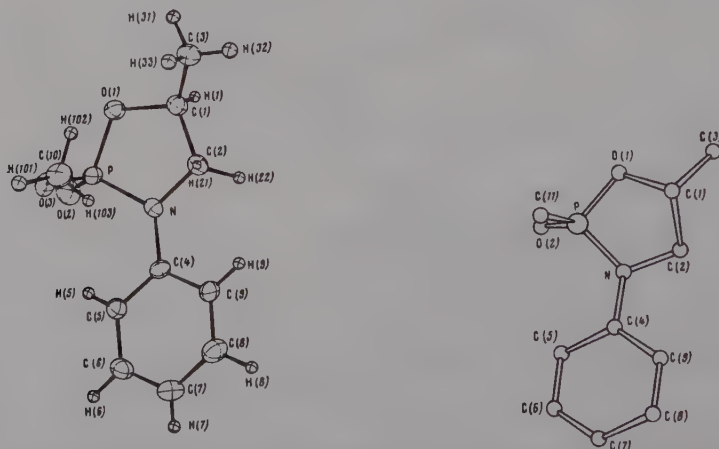


Fig. 1. Molecular structure of  $C_{10}H_{14}NO_3P$  (left), and  $C_{10}H_{14}NO_2P$  (without H atoms) (right).

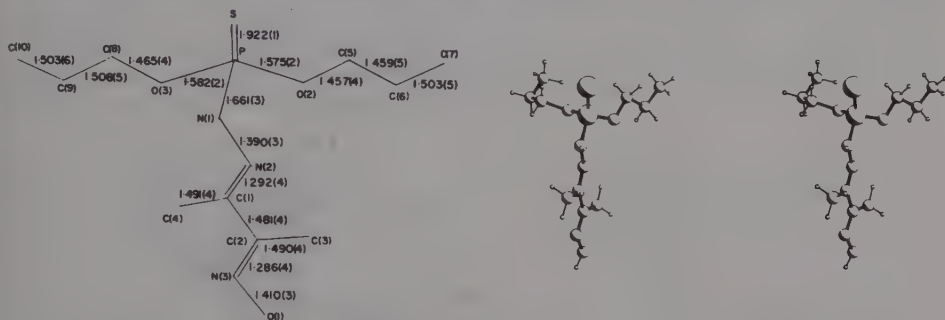
The five-membered heterocyclic rings in I and II have envelope conformations (Fig. 1). Atom C(1) is folded towards the oxo group in I and towards the methyl substituent at P in II. Bond distances (in I and II respectively) associated with the P atom are: P-N 1.649, 1.664, P-O(1) 1.572, 1.589, P-O(2) 1.448, 1.35, P-O(3) 1.573, P-C(11) 1.89 Å. The molecule II is disordered with the two enantiomeric forms statistically superimposed.

O,O-DIPROPYL-(E)-2-(1-METHYL-2-OXOPROPYLIDENE)PHOSPHOROHYDRAZIDOTHIOATE-(E)-OXIME  
 $C_{10}H_{22}N_3O_3PS$

M. ALAM, R. SANDUJA, M.B. HOSSAIN and D. van der HELM, 1982. J. Am. Chem. Soc., 104, 5232-5234.

Monoclinic,  $P2_1/c$ ,  $a = 11.255$ ,  $b = 17.694$ ,  $c = 8.214$  Å,  $\beta = 109.60^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.048$  for 2436 reflexions (at  $-135^\circ C$ ).

The geometry of this ichthyotoxic metabolite of Gymmodinium breve is shown in Fig. 1. Excluding the OPr groups, the molecule is planar within 0.10 Å. The P-N distance of 1.661(3) Å is somewhat longer than normal.



In the molecule (Fig. 1) the phosphetane ring is puckered with a dihedral angle of  $25.8(3)^\circ$ . Intermolecular contacts correspond to van der Waals interactions and as with other phosphetanes the crystal packing is somewhat loose.

METHYL 3-METHYL-1,7-DIOXO-1-PHOSPHA-2-OXATRICYCLO(4.3.1.0<sup>3,8</sup>)DECANE-6-CARBOXYLATE  
C<sub>11</sub>H<sub>15</sub>O<sub>5</sub>P

D.C. CRAIG, M.J. GALLAGHER and J. SUSSMAN, 1982. Cryst. Struct. Comm., 11, 2083-2086.

Orthorhombic, Pbc<sub>a</sub>,  $a = 11.493$ ,  $b = 20.540$ ,  $c = 9.684$  Å,  $D_m = 1.49$ ,  $Z = 8$ . Cu radiation,  $R = 0.038$  for 2067 reflexions.

The ring conformations (Fig. 1) are C1-C2-C3-C4-C5-C6 twist boat, P-C7-C6-C5-C4-C8 chair and P-O1-C1-C6-C7 envelope, with C7 the out-of-plane atom. The angle of phosphorus in the five-membered ring is smaller than expected ( $95.7(1)^\circ$ ), but the angle in the six-membered ring is normal ( $102.1(1)^\circ$ ).

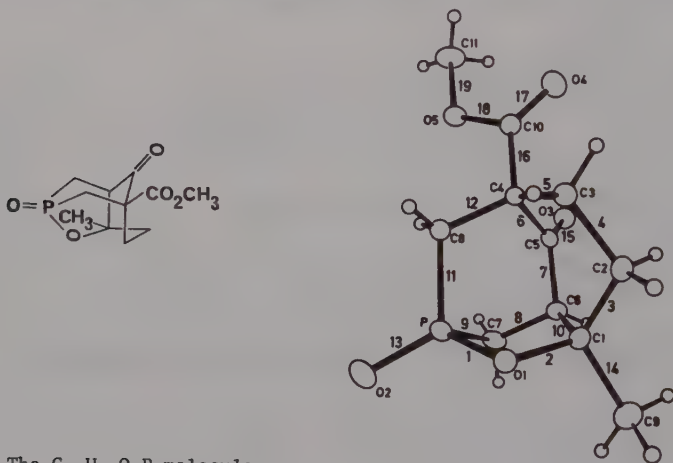


Fig. 1. The C<sub>11</sub>H<sub>15</sub>O<sub>5</sub>P molecule.

MONO-(-)- $\alpha$ -PHENETHYLAMMONIUM (+)-cis-(1S,2R)-(1,2-EPOXYPROPYL)PHOSPHONATE MONOHYDRATE  
C<sub>11</sub>H<sub>18</sub>NO<sub>3</sub>P·H<sub>2</sub>O C<sub>3</sub>H<sub>6</sub>O<sub>3</sub>P<sup>-</sup>, C<sub>8</sub>H<sub>12</sub>N<sup>+</sup>, H<sub>2</sub>O

A. PERALES, M. MARTÍNEZ-RIPOLL, J. FAYOS, C. VON CARSTENN-LICHTERFELDE and M. FERNANDEZ, 1982. Acta Cryst., B38, 2763-2764.

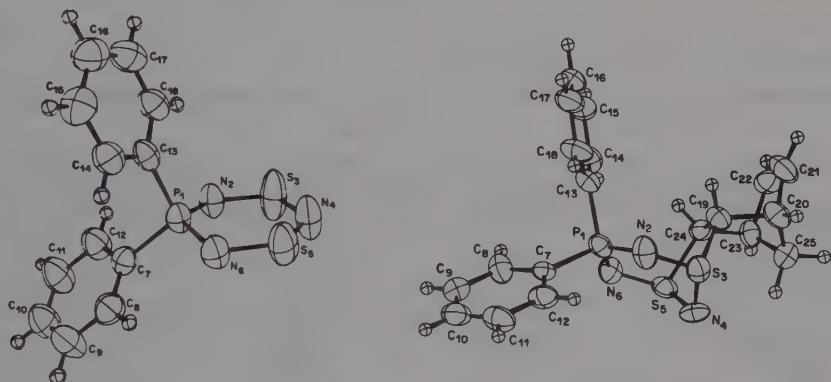
Monoclinic, P2<sub>1</sub>,  $a = 11.530$ ,  $b = 6.1490$ ,  $c = 10.199$  Å,  $\beta = 102.76^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.040$  for 1306 reflexions.

The X-ray intensities for the title compound were used to determine the absolute stereochemistry of both enantiomers of fosfomycin, (-)-(1R,2S)-(1,2-epoxypropyl)phosphonic acid. The active form is 1R,2S (1), the inactive form being 1S,2R. The crystal structure itself is not discussed.

1. Structure Reports, 44B, 613.

1,1-DIPHENYL-CYCLO-1 $\lambda^5$ -PHOSPHA-3,5-DITHIA-2,4,6-TRIAZENE $C_{12}H_{10}N_3PS_2$  (I) $(C_6H_5)_2PS_2N_3$ exo- $\beta$ -10,10-DIPHENYL-1 $\lambda^5$ -PHOSPHA-1,8-DITHIA-9,11,12-TRIAZA-TETRACYCLO[6.3.1.1<sup>1,8</sup>-.0<sup>3,6</sup>]TRIDECA-1(12),4,8,10-TETRAENE TOLUENE $C_{12}H_{10}N_3PS_2 \cdot C_7H_8$  $(C_6H_5)_2PS_2N_3 \cdot C_7H_8$ N. BUFORD, T. CHIVERS, A.W. CORDES, W.G. LAIDLAW, M.C. NOBLE, R.T. OAKLEY and P.N. SWEPSTON, 1982. J. Am. Chem. Soc., 104, 1282-1290.I. Monoclinic,  $P2_1/c$ ,  $a = 8.305$ ,  $b = 13.183$ ,  $c = 12.487$  Å,  $\beta = 105.53^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.047$  for 1471 reflexions.II. Triclinic,  $P1$ ,  $a = 9.239$ ,  $b = 6.284$ ,  $c = 9.314$  Å,  $\alpha = 76.98$ ,  $\beta = 119.98$ ,  $\gamma = 110.08^\circ$ ,  $Z = 1$ . Cu radiation,  $R = 0.046$  for 1583 reflexions.

The two molecules are shown in Fig. 1. I contains a six-membered  $PS_2N_3$  ring in which the NSNSN fragment is planar to within 0.05 Å, while the P atom is 0.284(1) Å out of this plane. The S-N distances are within the range 1.560(3)-1.583(5) Å and the mean P-N distance is 1.623(4) Å. The N-P-N and C-P-C angles are 115.8(2) and 107.1(2)° respectively. The structure of II shows that the addition of norbornadiene to I occurs in a 1,3 fashion across the two S atoms to give the exo- $\beta$  isomer of II. As a result of the addition, the central N atom of the NSNSN unit is displaced by 0.817(6) Å from the plane of the central  $S_2N_2$  unit, which is planar to within 0.01 Å. The S-N bond lengths are 1.604(6)-1.655(7) Å, the lengthening being attributed primarily to the rupture of the  $\pi$  system.

Fig. 1. The structures of  $(C_6H_5)_2PS_2N_3$  (left) and  $(C_6H_5)_2PS_2N_3 \cdot C_7H_8$  (right).

## O,O-DIETHYL-N-CHLOROPHOSPHORAMIDATE

 $C_{12}H_{11}ClNO_3P$  $(C_6H_5O)_2P(O)NHC1$ K. DREWELIES and H. PRITZKOW, 1982. Z. Naturforsch., 37b, 1402-1405.Monoclinic,  $P2_1/a$ ,  $a = 11.033$ ,  $b = 13.674$ ,  $c = 9.912$  Å,  $\beta = 120.65^\circ$ ,  $D_m = 1.465$ ,  $Z = 4$ . Mo radiation,  $R = 0.055$  for 2539 reflexions.

The molecules (Fig. 1) are joined in pairs across centres of symmetry by two N-H...O(3) hydrogen bonds (2.85(1) Å). Principal bond lengths are: P-O(1) 1.574(1), P-O(2) 1.566(1), P-O(3) 1.456(2), P-N 1.637(2), N-Cl 1.705(2) Å.



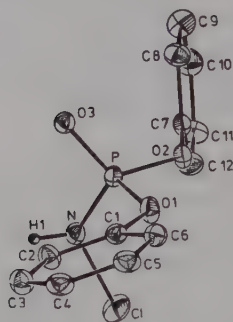


Fig. 1. The  $C_{12}H_{11}ClNO_3P$  molecule.

TRIS( $\eta^1$ -1-PYRROLYL)PHOSPHINE  
 $C_{12}H_{12}N_3P$  (I)

$(\eta^1-C_4H_4N)_3P$

TRIS( $\eta^1$ -1-PYRROLYL)ARSINE  
 $C_{12}H_{12}AsN_3$  (II)

$(\eta^1-C_4H_4N)_3As$

J.L. ATWOOD, A.H. COWLEY, W.E. HUNTER and S.K. MEHROTRA, 1982. *Inorg. Chem.*, **21**, 1354-1356.

I. Monoclinic,  $P2_1/n$ ,  $a = 12.428$ ,  $b = 5.858$ ,  $c = 17.071$  Å,  $\beta = 98.48^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.067$  for 845 reflexions.

II. Orthorhombic,  $P2_12_12_1$ ,  $a = 7.251$ ,  $b = 8.070$ ,  $c = 21.085$  Å,  $Z = 4$ . Mo radiation,  $R = 0.073$  for 883 reflexions.

The two molecules are very similar (Fig. 1), the pyrrolyl ligands being  $\eta^1$ -bonded in case. The P-N distances are 1.677(8)-1.710(8) Å and As-N, 1.84(2)-1.87(2) Å. The P and As coordination is pyramidal with N-P-N 100.1(4)-100.7(4)° and N-As-N 94.7(7)-98.9(7)°.

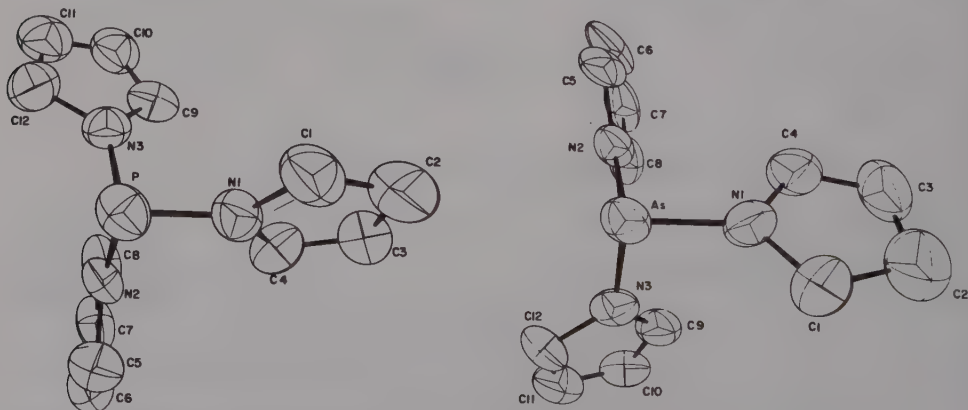


Fig. 1. The  $(\eta^1-C_4H_4N)_3E$  molecules:  $E = P$  (left) and  $As$  (right).

BIS(ANILINO)PHOSPHINE OXIDE  
 $C_{12}H_{13}N_2OP$

$(C_6H_5NH)_2P(O)H$

M.L. THOMPSON, R.C. HALTIWANGER, A. TARASSOLI, D.E. COONS, and A.D. NORMAN, 1982. *Inorg. Chem.*, **21**, 1287-1291.

I. Monoclinic,  $C2/c$ ,  $a = 17.929$ ,  $b = 5.232$ ,  $c = 24.804$  Å,  $\beta = 103.59^\circ$ ,  $D_m = 1.36$ ,  $Z = 8$ . Mo radiation,  $R = 0.039$  for 1316 reflexions.

II. Orthorhombic,  $Pnma$ ,  $a = 7.702$ ,  $b = 26.723$ ,  $c = 5.558$  Å,  $D_m = 1.34$ ,  $Z = 4$ . Mo radiation,  $R = 0.049$  for 652 reflexions.

Molecules of both forms are shown in Fig. 1, the orthorhombic (form II) molecule displaying exact mirror symmetry. In both forms molecules are linked by intermolecular N-H...O hydrogen bonds, pairwise with each pair bonded to another in form I and in a linear head-to-tail fashion in form II. P-O, mean P-N, N-P-N, and mean O-P-N are (respectively): 1.475(2), 1.639(3) Å, 105.6(15), 114.0(14)° for I; 1.471(4), 1.642(4) Å, 100.4(3), 116.3(2)° for II.

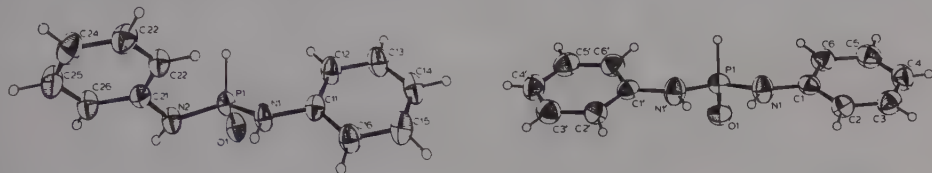


Fig. 1. The monoclinic (left) and orthorhombic forms of the  $C_{12}H_{13}N_2OP$  molecule.

#### 4,5,7,8-TETRAMETHYL-1,2-DIPHOSPHATRICYCLO[5.2.1]DECA-4,8-DIENE $C_{12}H_{18}P_2$

G. de LAUZON, G. CHARRIER, H. BONNARD, F. MATHEY, J. FISCHER and A. MITSCHLER, 1982. J. Chem. Soc. Chem. Commun., 1272-1273.

Monoclinic,  $P2_1$ ,  $a = 9.598$ ,  $b = 8.420$ ,  $c = 7.487$  Å,  $\beta = 93.98^\circ$ ,  $D_m = 1.24$ ,  $Z = 2$ . Cu radiation,  $R = 0.049$  for 1756 reflexions.

The dimer (Fig. 1) has the endo structure with long, weak P-P (2.239(1)) and C-C bridges (1.577(5) Å) between the two phosphole units. Other bond distances are: P(1)-C(1) 1.844(4), P(1)-C(4) 1.812(4), P(2)-C(7) 1.877(4), P(2)-C(10) 1.849(4), C(1)-C(2) 1.542(5), C(2)-C(3) 1.518(5), C(3)-C(4) 1.337(5), C(7)-C(8) 1.508(5), C(8)-C(9) 1.319(5), C(9)-C(10) 1.515(6) Å. Bond angles are: C(1)-P(1)-C(4) 87.6(2), C(7)-P(2)-C(10) 92.8(1), C(1)-P(1)-P(2) 86.0(1), C(4)-P(1)-P(2) 97.5(1), P(1)-P(2)-C(7) 93.3(1), P(1)-P(2)-C(10) 104.5(1), P(1)-C(1)-C(2) 102.6(2), C(1)-C(2)-C(7) 104.2(3), P(2)-C(7)-C(8) 103.6(2), P(2)-C(7)-C(2) 109.8(2) and C(2)-C(7)-C(8) 116.6(3)°.

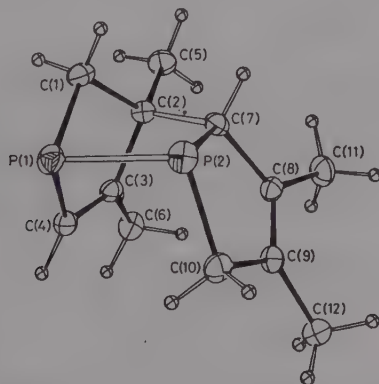
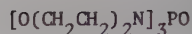
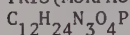


Fig. 1.  $C_{12}H_{18}P_2$ : ORTEP plot of the dimer.

## TRIS(MORPHOLINO)PHOSPHINE OXIDE



C. RØMMING and J. SONGSTAD, 1982. *Acta Chem. Scand.*, **A36**, 665-671.

Monoclinic,  $\text{P}2_1/\text{n}$ ,  $a = 9.107$ ,  $b = 11.145$ ,  $c = 14.605 \text{ \AA}$ ,  $\beta = 97.03^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.045$  for 4151 reflexions (at  $-150^\circ\text{C}$ ).

One of the morpholino groups is disordered (Fig. 1) and in order to obtain satisfactory resolution the atoms of this ring together with the P atom and the attached O atom had to be split. The two "half" morpholino rings are inversion forms and are oriented in such a way that the direction of the N lone pair is approximately retained after the inversion. The average P-N bond length is  $1.66 \text{ \AA}$  and the sum of the N bond angles ranges from  $349.1$  to  $358.6^\circ$ .

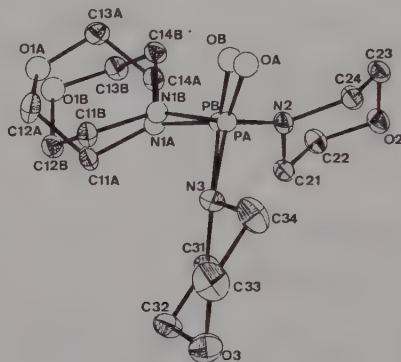
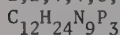


Fig. 1. Molecular structure of  $\text{C}_{12}\text{H}_{24}\text{N}_3\text{O}_4\text{P}$ .

## 2,2,4,4,6,6-HEXA(1-AZIRIDINYL)CYCLOTRI(PHOSPHAZENE)



T.S. CAMERON, J.-F. LA BARRE and M. GRAFFEUIL, 1982. *Acta Cryst.*, **B38**, 2000-2004.

I. Orthorhombic,  $\text{P}2_12_12_1$ ,  $a = 7.981$ ,  $b = 13.641$ ,  $c = 16.589 \text{ \AA}$ ,  $Z = 4$ . Mo radiation,  $R = 0.027$  for 1030 reflexions.

II. Monoclinic,  $\text{P}2_1/\text{c}$ ,  $a = 9.626$ ,  $b = 8.099$ ,  $c = 23.67 \text{ \AA}$ ,  $\beta = 101.39^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.053$  for 998 reflexions.

In both crystal forms the molecules have almost identical conformations and geometries with the geminal aziridinyl groups adopting the trans configuration (Fig. 1). Atoms N(1), N(2), N(3) are  $0.17$ ,  $0.07$ ,  $0.01(5) \text{ \AA}$  respectively from the  $\text{P}_3$  plane giving a slight crown configuration. Mean P-N distances are  $1.596(3)$ , endocyclic and  $1.676(4)$ , exocyclic with mean C-C  $1.465(4) \text{ \AA}$ . Five of the pyramidal aziridinyl groups have very similar conformations with mean C-N  $1.466(7) \text{ \AA}$ ; in the N(21)-C(211)-C(212) group C-N distances are  $1.431(8)$  and  $1.461(8) \text{ \AA}$ .

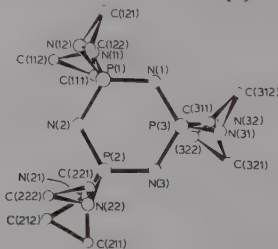
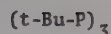
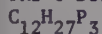


Fig. 1. The  $\text{P}_3\text{N}_3[\text{N}(\text{CH}_2)_2]_6$  molecule.

TRI-*t*-BUTYLCYCLOTRIPHOSPHANE

J. HAHN, M. BAUDLER, C. KRUGER and Y.-H. TSAY, 1982. *Z. Naturforsch.*, **37b**, 797-805.

Triclinic,  $P\bar{1}$ ,  $a = 6.256$ ,  $b = 17.415$ ,  $c = 17.806$  Å,  $\alpha = 62.071$ ,  $\beta = 79.885$ ,  $\gamma = 79.664^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.083$  for 5834 reflexions.

The steric interactions between the two large *t*-butyl groups in a *cis* arrangement (Fig. 1) cause a significant lengthening of the P-P bond (2.215(2) Å) compared with the P-P bonds where the substituent arrangement is *trans* (2.186(1) Å). The P-P-C bond angles are also greatly enlarged (123.3(1)° compared to 106.9(1)°).

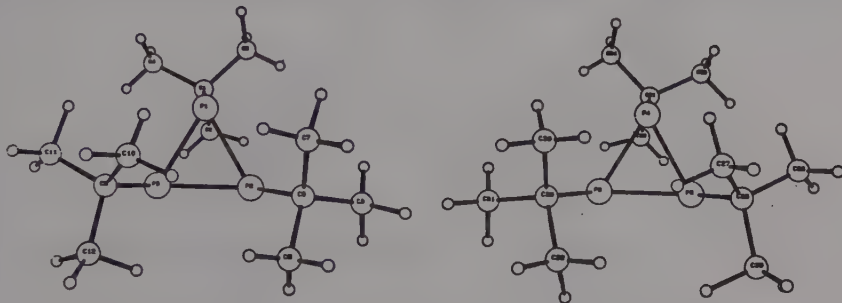
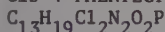


Fig. 1. The two crystallographically independent molecules of  $C_{12}H_{27}P_3$ .

## cis-4-PHENYLCYCLOPHOSPHAMIDE



V.L. HIMES, A.D. MIGHELL, J.K. STALICK and G. ZON, 1982. *Acta Cryst.*, **B38**, 1009-1012.

Monoclinic,  $P2_1/c$ ,  $a = 8.000$ ,  $b = 10.279$ ,  $c = 20.003$  Å,  $\beta = 102.12^\circ$ ,  $D_m = 1.39$ ,  $Z = 4$ . Mo radiation,  $R = 0.037$  for 1206 reflexions.

The molecule (Fig. 1) exists in a chair conformation with the phenyl substituent and the phosphoryl O atom in equatorial positions. The crystal structure consists of centrosymmetric dimers (Fig. 1) linked by hydrogen bonds between N-H and O=P (N...O 2.837 Å).

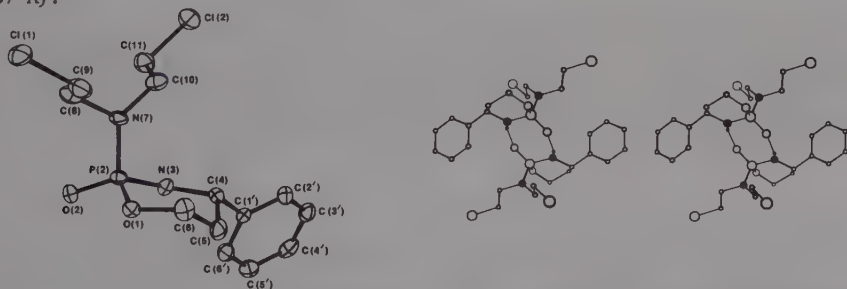
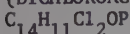


Fig. 1.  $C_{13}H_{19}Cl_2N_2O_2P$ : conformation of the molecule (left) and stereoscopic view of the centrosymmetric dimer formed by N-H...O=P hydrogen bonds (right).

## (DICHLOROACETYL) DIPHENYLPHOSPHINE



E. LINDNER, M. STEINWAND and S. HOEHNE, 1982. *Chem. Ber.*, **115**, 2181-2191.

Monoclinic,  $P2_1/c$ ,  $a = 11.134$ ,  $b = 13.456$ ,  $c = 18.110$  Å,  $\beta = 96.76^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.034$  for 2157 reflexions (at  $-110^\circ\text{C}$ ).

The P atom (Fig. 1) has trigonal pyramidal coordination with P-C(1) 1.881(5), P-C(phenyl) 1.826(5) Å. The C(2)-Cl(2) bond is almost eclipsed by the C(1)-O bond (dihedral angle  $3.0^\circ$ ).

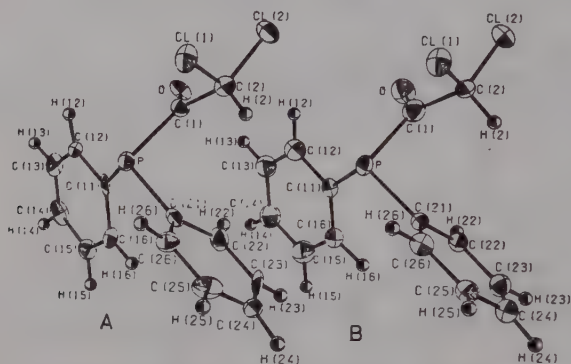


Fig. 1. The two crystallographically independent  $(\text{C}_6\text{H}_5)_2\text{P(Se)COCHCl}_2$  molecules.

P,P-DIMORPHOLINOPHENYLPHOSPHINE SELENIDE

$\text{C}_{14}\text{H}_{21}\text{N}_2\text{O}_2\text{PSe}$  (I)

$[\text{O}(\text{CH}_2\text{CH}_2)_2\text{N}]_2\text{P(Se)}\text{C}_6\text{H}_5$

P,P-DIPHENYLMORPHOLINOPHOSPHINE SELENIDE

$\text{C}_{16}\text{H}_{18}\text{NOPSe}$  (II)

$\text{O}(\text{CH}_2\text{CH}_2)_2\text{NP(Se)}(\text{C}_6\text{H}_5)_2$

K. MAARTMANN-MOE, C. RØMMING and J. SONGSTAD, 1982. Acta Chem. Scand., **A36**, 757-765.

I. Monoclinic,  $P2_1/c$ ,  $a = 10.266$ ,  $b = 17.286$ ,  $c = 9.706$  Å,  $\beta = 112.06^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.033$  for 2632 reflexions.

II. Orthorhombic,  $P2_1cn$ ,  $a = 7.874$ ,  $b = 14.099$ ,  $c = 14.539$  Å,  $Z = 4$ . Mo radiation,  $R = 0.032$  for 1090 reflexions.

The sums of the bond angles at the N atoms are  $342.9$  and  $348.2^\circ$  in I and  $344.4^\circ$  in II. In I, the lone pair of the N atom of highest p-character is antiperiplanar to the P-Se bond; the lone pair of the other N atom is roughly orthogonal to the P-Se bond. In II the lone pair of the N atom is strictly antiperiplanar to the P-Se bond (Fig. 1). The P-N bond lengths are 1.669 and 1.662 in I and 1.672 Å in II. The P-Se bond lengths are 2.097 and 2.102 Å in I and II respectively.

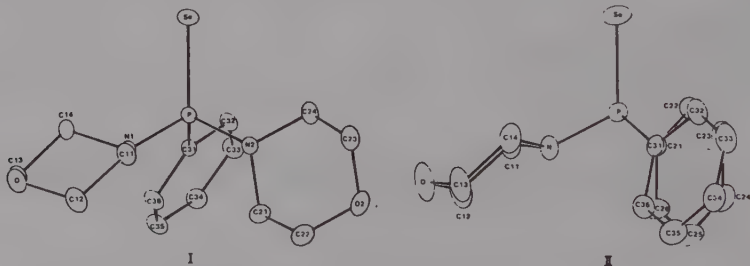


Fig. 1. Molecular structures of  $\text{C}_{14}\text{H}_{21}\text{N}_2\text{O}_2\text{PSe}$  (I) (left), and  $\text{C}_{16}\text{H}_{18}\text{NOPSe}$  (II) (right).

TRIETHYLAMMONIUM BIS(C,C-DIMETHYL-HYDROXY-ACETOXY)-SPIROPHOSPHORANO-DITHIOFORMATE  
 $C_{15}H_{28}NO_6PS_2$   $Et_3NH^+, C_9H_{12}O_6PS_2^-$

A. DUBOURG, R. ROQUES, G. GERMAIN, J.P. DECLERCQ, A. MUNOZ, A. KLAÉBÉ, B. GARRIGUES and R. WOLF, 1982. Phosphorus and Sulfur, 14, 121-129.

Orthorhombic,  $Pn2_1a$ ,  $a = 12.239$ ,  $b = 11.169$ ,  $c = 15.424$  Å,  $Z = 4$ . Cu radiation,  $R = [?]$  for 1450 reflexions.

The phosphorus atom has trigonal-bipyramidal geometry (Fig. 1) with axial P-O 1.731 and 1.738(4), equatorial P-O 1.577 and 1.582(5), P-C 1.836(5), C-S (average) 1.66 Å and S-C-S 129.3°.

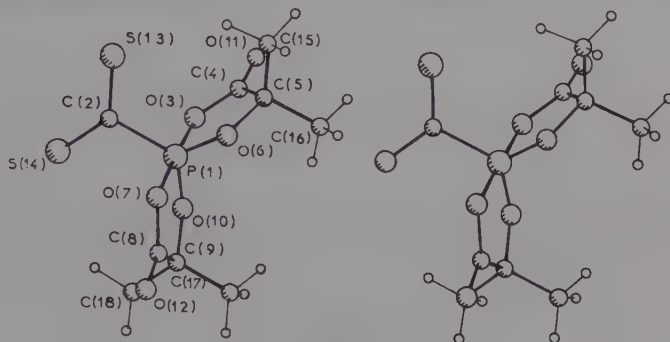
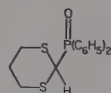


Fig. 1. A stereoview of the  $C_9H_{12}O_6PS_2^-$  anion.

2-[1,3]DITHIANYL-DIPHENYLPHOSPHINE OXIDE  
 $C_{16}H_{17}OPS_2$



E. JUARISTI, L. VALLE, C. MORA-UZETA, B.A. VALENZUELA, P. JOSEPH-NATHAN and M.F. FREDRICH, 1982. J. Org. Chem., 47, 5038-5039.

Monoclinic,  $P2_1/c$ ,  $a = 12.304$ ,  $b = 5.6311$ ,  $c = 22.826$  Å,  $\beta = 99.84^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.049$  for 2004 reflexions.

The heterocyclic six-membered ring exists in a chair conformation with the substituent at C(1) being axial. The plane containing S(1), S(2), C(2) and C(4) bisects that passing through C(2), C(3) and C(4) at  $58.1^\circ$  and the S(1), C(1), S(2) plane at  $54.1^\circ$ . The two latter planes are almost parallel, forming an angle of  $4.1^\circ$ . The oxygen atom is centered above the heterocyclic ring and the two phenyl ring planes bisect at  $85.8^\circ$ .

cis-1,2,4,5-TETRAMETHYL-3,6-DIPHENOXY-1,2,4,5-TETRAAZA-3,6-DIPHOSPHACYCLOHEXANE-3,6-DISULPHIDE

$C_{16}H_{22}N_4O_2P_2S_2$

U. ENGELHARDT and H. VIERTTEL, 1982. Acta Cryst., B38, 1972-1975.

Orthorhombic,  $Pbca$ ,  $a = 15.655$ ,  $b = 30.984$ ,  $c = 8.780$  Å,  $Z = 8$ . Cu radiation,  $R = 0.047$  for 2484 reflexions.

The heterocyclic ring has a twist conformation (Fig. 1); the PNN angles are in the range  $113.7(3)$  to  $115.1(4)$  and the NPN angles are  $99.6(2)$  and  $100.7(2)^\circ$ . Mean bond lengths are: P-N 1.655(6), N-N 1.413(2), P-S 1.910(3), P-O 1.595(1) and C-O 1.410(3) Å.



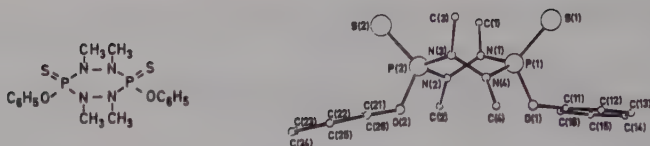
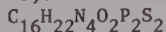


Fig. 1. The structure of the  $C_{16}H_{22}N_4O_2P_2S_2$  molecule.

trans-1,2,4,5-TETRAMETHYL-3,6-DIPHENOXY-1,2,4,5-TETRAAZA-3,6-DIPHOSPHACYCLOHEXANE  
3,6-DISULPHIDE



U. ENGELHARDT and H. VIERTTEL, 1982. *Acta Cryst.*, **B38**, 3049-3052.

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.530$ ,  $b = 8.081$ ,  $c = 35.522$  Å,  $Z = 4$ . Cu radiation,  $R = 0.047$  for 1776 reflexions.

The heterocycle (Fig. 1) has a twist conformation; the PNN angles range from  $111.9(5)$  to  $116.4(5)^\circ$ , and the NPN angles are  $99.3(3)$  and  $101.0(3)^\circ$ . Mean bond distances are: P-N  $1.648(6)$ , N-N  $1.40(1)$ , P-S  $1.910(3)$ , P-O  $1.597(5)$ , O-C  $1.384(8)$  Å.

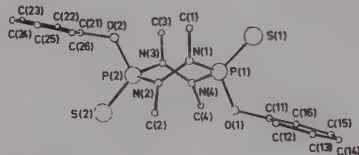
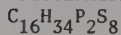


Fig. 1. The  $C_{16}H_{22}N_4O_2P_2S_2$  molecule.

cis-2,10-DI(t-BUTYL)-1,3,6,9,11,14-HEXATHIA-2,10-DIPHOSPHACYCLOHEXADECANE 2,10-DISULPHIDE



A. GRAND and J. MARTIN, 1982. *Acta Cryst.*, **B38**, 3052-3055.

Monoclinic,  $P2_1/c$ ,  $a = 10.054$ ,  $b = 11.929$ ,  $c = 21.861$  Å,  $\beta = 97.38^\circ$ ,  $D_m = 1.36$ ,  $Z = 4$ . Mo radiation,  $R = 0.064$  for 3009 reflexions (at 213 K).

The molecule adopts an asymmetric conformation (Fig. 1) which does not belong to conventional forms; there is also a slight difference between bond lengths and angles in the two parts of the molecule.

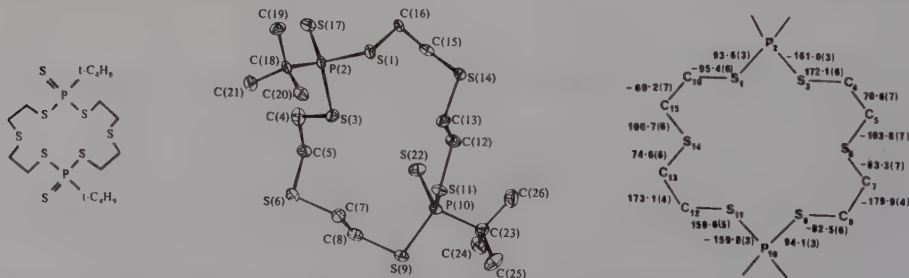
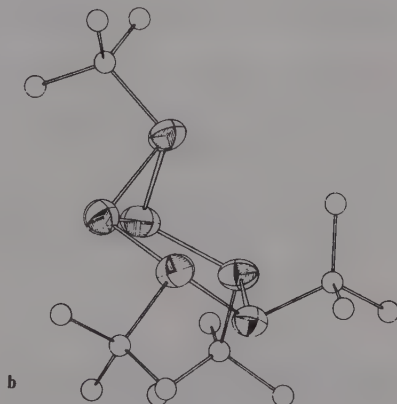
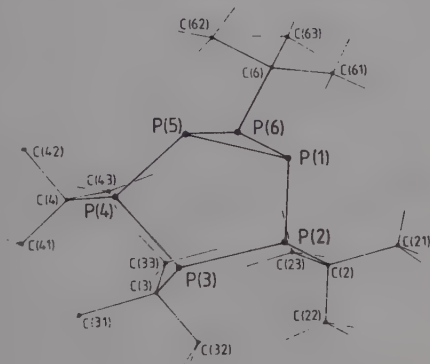


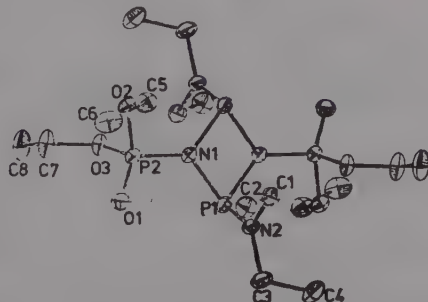
Fig. 1. A view of  $C_{16}H_{34}P_2S_8$  and ring torsion angles.

2,3,4,6-TETRA-*t*-BUTYLBICYCLO[3.1.0]HEXAPHOSPHANE $C_{16}H_{36}P_6$ *t*-Bu<sub>4</sub>P<sub>6</sub>K.-F. TEBBE and T. HEINLEIN, 1982: *Z. Krist.*, **160**, 285-298.Monoclinic,  $P2_1/n$ ,  $a = 9.120$ ,  $b = 13.838$ ,  $c = 19.514$  Å,  $\beta = 90.93^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.045$  for 860 reflexions.

The molecule (Fig. 1) has a chair-like skeleton, with the three-membered ring almost perpendicular to the five-membered ring which is an unusually flat, only slightly twisted envelope. Some bond lengths are mean endocyclic P-P distances 2.199 Å (five-membered ring) 2.183 Å (three-membered ring), common edge P-P 2.197 Å. The *t*-butyl groups are all-*trans*. The mean P-C and C-C distances are 1.904 and 1.524 Å.

Fig. 1. Two views of  $C_{16}H_{36}P_6$ .trans-1,3-BIS(DIETHOXYPHOSPHORYL)-2,4-BIS(DIETHYLAMINO)-1,3,2,4-DIAZA- $\lambda^3, \lambda^3$ -DIPHOSPHETIDINE $C_{16}H_{40}N_4O_6P_4$ H. RICHTER, E. FLUCK, H. RIFFEL and H. HESS, 1982. *Z. Anorg. Chem.*, **486**, 177-186.Monoclinic,  $C2/c$ ,  $a = 21.049$ ,  $b = 9.755$ ,  $c = 13.026$  Å,  $\beta = 102.79^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.058$  for 2978 reflexions.

The molecule (Fig. 1) has  $C_i$  symmetry; the two N atoms have a planar trigonal configuration while the P(V) and P(III) atoms are tetrahedrally and trigonal-pyramidally coordinated respectively (P(2)-N(1) 1.650(2), O(1) 1.468(2), O(2) 1.590(2), O(3) 1.582(2); P(1)-N(1) 1.749(2), N(1') 1.760(2), N(2) 1.649(2) Å).

Fig. 1. The centrosymmetric  $C_{16}H_{40}N_4O_6P_4$  molecule.

trans-1,3-BIS(DIETHOXYPHOSPHORYL)-2,4-BIS(DIETHYLAMINO)-2-OXO-1,3,2,4-DIAZA- $\lambda^5, \lambda^3$ -DIPHOSPHETIDINE

$C_{16}H_{40}N_4O_7P_4$  (I)

trans-1,3-BIS(DIETHOXYPHOSPHORYL)-2,4-BIS(DIETHYLAMINO)-2-THIO-1,3,2,4-DIAZA- $\lambda^5, \lambda^3$ -DIPHOSPHETIDINE

$C_{16}H_{40}N_4O_6P_4S$  (II)

trans-1,3-BIS(DIETHOXYPHOSPHORYL)-2,4-BIS(DIETHYLAMINO)-2,4-DITHIO-1,3,2,4-DIAZA- $\lambda^5, \lambda^5$ -DIPHOSPHETIDINE

$C_{16}H_{40}N_4O_6P_4S_2$  (III)

E. FLUCK, H. RICHTER, H. RIFFEL and H. HESS, 1982. Phosphorus and Sulfur, 14, 87-89.

I. Monoclinic,  $C2/c$ ,  $a = 20.995$ ,  $b = 9.797$ ,  $c = 13.090$  Å,  $\beta = 103.00^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.048$  for 1403 reflexions.

II. Monoclinic,  $P2_1/a$ ,  $a = 13.622$ ,  $b = 11.904$ ,  $c = 16.517$  Å,  $\beta = 91.73^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.090$  for 3526 reflexions.

III. Monoclinic,  $P2_1/n$ ,  $a = 10.884$ ,  $b = 11.692$ ,  $c = 11.018$  Å,  $\beta = 98.98^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.064$  for 2867 reflexions.

The paper lists coordinates and dimensions for the molecules I-III (Fig. 1). There is little crystallographic discussion. Molecule III lies about an inversion centre.

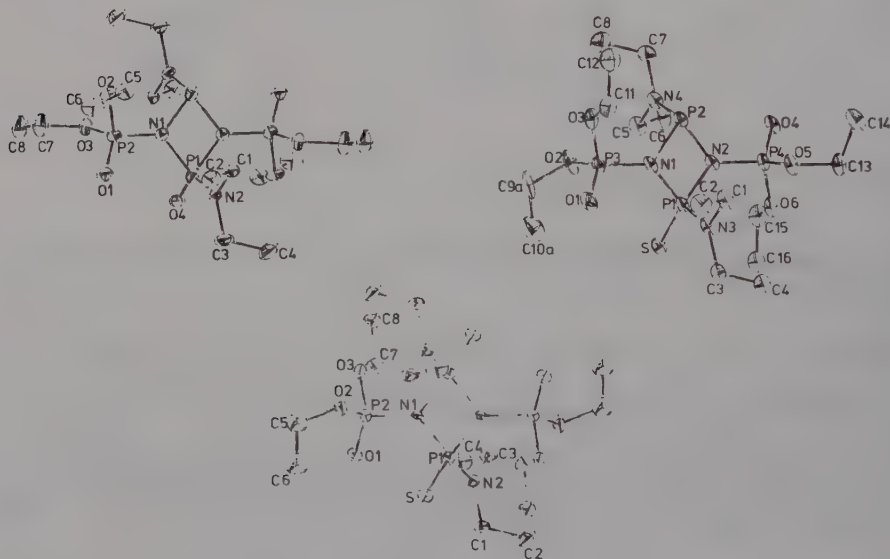


Fig. 1. Views of the 2-oxo- (I) (top right), 2-thio (II) (top left) and 2,4-dithio (III) (bottom) diphosphetidines.

TETRAKIS(DIETHYLAMINO)DITHIOXODI- $\lambda^5$ -PHOSPHANE

$C_{16}H_{40}N_4P_2S_2$

$((Et)_2N)_2P(S)-P(S)(N(Et)_2)_2$

D. TROY, J.P. LEGROS and G.P. McQUILLAN, 1982. Inorg. Chim. Acta, 58, 217-225.

Monoclinic,  $P2_1/n$ ,  $a = 9.208$ ,  $b = 12.859$ ,  $c = 10.397$  Å,  $\beta = 97.56^\circ$ ,  $D_m = 1.06$ ,  $Z = 2$ . Mo radiation,  $R = 0.096$  for 1090 reflexions.

The molecule consists of two  $(Et_2N)_2P(S)$  moieties related by a crystallographic centre of symmetry in the middle of the P-P bond (Fig. 1). The N atoms are in a planar environment with P-N(1)-C(12) and P-N(1)-C(11) angles of 126 and 119° respectively (corresponding angles for N(2) are 117 and 135°, the large angular deviation from normal being caused by disorder in the methylene carbon atoms). The P-P' and the P-S distances are respectively 2.244 and 1.956 Å while those of P-N(1) and P-N(2) are 1.644 and 1.667 Å respectively.

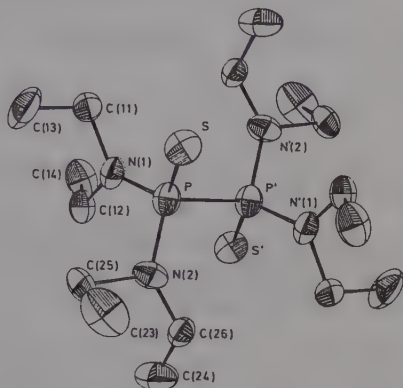


Fig. 1. Molecular structure of  $C_{16}H_{40}N_4P_2S_2$  (disordered carbon atoms are not shown).

O-( $\beta$ -DIMETHYLAMINOETHYL)PHENYL- $\alpha$ -HYDROXYCYCLOHEXYLPHOSPHINATE IODIDE (high melting)  
 $C_{17}H_{29}INO_3P$   $[C_6H_5(C_6H_{10}OH)P(O)O(CH_2)_2N(CH_3)_3]^+I^-$

N.A. KARDANOV, V.V. TKACHEV, L.O. ATOVMYAN, N.N. GODOVIKOV and M.I. KABACHNIK, 1982.  
 Zh. Strukt. Khim., 23-2, 80-88 [J. Struct. Chem., 23, 235-241].

Monoclinic,  $P2_1/b$ ,  $a = 14.294$ ,  $b = 9.253$ ,  $c = 15.440$  Å,  $\gamma = 90.05^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.088$  for 1830 reflexions.

The structure of the cation, which has the  $\phi^5$  conformation, is shown in Fig. 1. The other polymorphic modifications differ in mutual orientations of the P=O and C-OH groups. Bond lengths ( $\sigma = 0.01$ - $0.03$  Å) are normal.

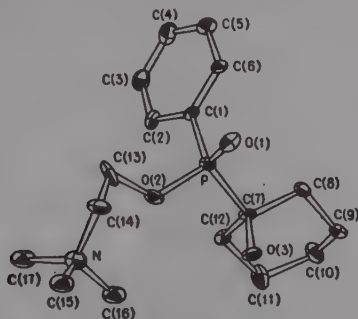
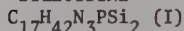
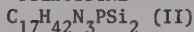


Fig. 1. The  $[C_6H_5(C_6H_{10}OH)P(O)O(CH_2)_2N(CH_3)_3]^+$  cation.

(E)-2-t-BUTYLTRIMETHYLSILYLAMINO-1,3-DI-t-BUTYL-4,4-DIMETHYL-1-3,2λ<sup>3</sup>-DIAZAPHOSPHASILETIDINE



(Z)-2-t-BUTYLTRIMETHYLSILYLAMINO-1,3-DI-t-BUTYL-4,4-DIMETHYL-1-3,2λ<sup>3</sup>-DIAZAPHOSPHASILETIDINE



O.J. SCHERER, M. PÜTTMANN, C. KRÜGER and G. WOLMERSHÄUSER, 1982. Chem. Ber., 115, 2076-2124.

I. Orthorhombic, Pbc<sub>a</sub>, a = 11.105, b = 28.641, c = 15.153 Å, Z = 8. Mo<sup>i</sup> radiation, R = 0.056 for 2567 reflexions.

II. Monoclinic, P2<sub>1</sub>/n, a = 10.888, b = 19.098, c = 11.463 Å, β = 91.97°, Z = 4. Mo radiation, R = 0.058 for 1764 reflexions.

The structures were established as those shown in Fig. 1. Principal bond lengths are: N(1)-Si(1) 1.760(3) (1.780(4)), N(1)-C(1) 1.531(4) (1.516(6)), N(1)-P 1.721(2) (1.712(4)), P-N(2,3) 1.740(3) (1.742(4)), Si(2)-N(2,3) 1.730(3) (1.731(4)) Å (the bond lengths for the Z-form are in brackets).

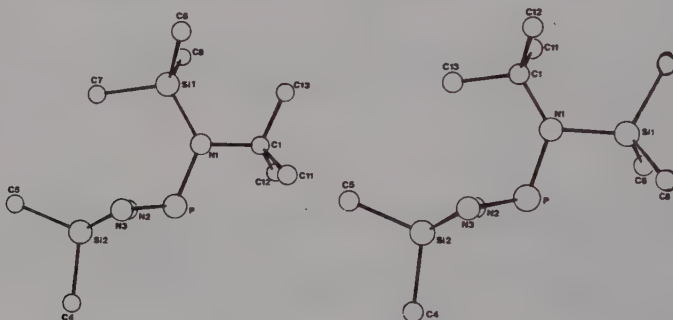
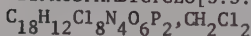


Fig. 1. The E (left) and Z (right) isomers of  $C_{17}H_{42}N_3PSi_2$ .

P,P'-BIS(TETRACHLOROCATECHOLATO-O,O')-2,4,6,8-TETRAMETHYL-2,4,6,8-TETRAAZA-1,5-DIPHOSPHABICYCLO[3.3.0]OCTANE-3,7-DIONE DICHLOROMETHANE SOLVATE



H.W. ROESKY, D. AMIRZADEH-ASL and W.S. SHELDRIK, 1982. J. Am. Chem. Soc., 104, 2919-2920.

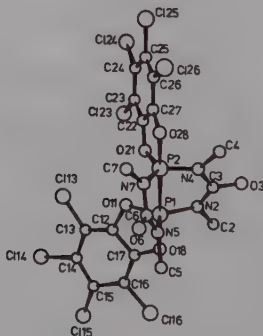


Fig. 1. The  $C_{18}H_{12}Cl_8N_4O_6P_2$  molecule.

Triclinic,  $P\bar{1}$ ,  $a = 12.429$ ,  $b = 15.954$ ,  $c = 8.145$  Å,  $\alpha = 100.46$ ,  $\beta = 100.77$ ,  $\gamma = 75.07^\circ$ ,  $D_m = 1.77$ ,  $Z = 2$ . Cu radiation,  $R = 0.061$  for 2472 reflexions.

The molecule (Fig. 1) contains an axial  $\lambda^5P-\lambda^5P$  bond of  $2.256(3)$  Å. Other bond lengths involving the trigonal bipyramidal P atoms are: P-O(ax.)  $1.714(6)$  and  $1.723(6)$ , P-O(eq.)  $1.675(5)$  and  $1.669(5)$ , P-N(eq.)  $1.682(6)$ - $1.693(6)$  Å.

# TRIANILINOPHOSPHINE

$C_{18}H_{18}N_3P$  (I)

$(C_6H_5NH)_3P$

# PHENYLBIS(DIANILINOPHOSPHINO)AMINE DIETHYLETHER (1:1)

$C_{30}H_{29}N_5P_2 \cdot C_4H_{10}O$  (II)

$[(C_6H_5NH)_2P]_2NC_6H_5 \cdot (C_2H_5)_2O$

A. TARASSOLI, R.C. HALTIWANGER and A.D. NORMAN, 1982. *Inorg. Chem.*, **21**, 2684-2690.

I. Hexagonal,  $P6_3$ ,  $a = 11.449$ ,  $c = 7.488$  Å,  $D_m = 1.21$ ,  $Z = 2$ . Mo radiation,  $R = 0.052$  for 442 reflexions.

II. Monoclinic,  $P2_1/n$ ,  $a = 9.193$ ,  $b = 21.154$ ,  $c = 16.624$  Å,  $\beta = 96.64^\circ$ ,  $D_m = 1.25$ ,  $Z = 4$ . Mo radiation,  $R = 0.075$  for 1403 reflexions.

The two structures are shown in Fig. 1. I has exact  $C_3$  symmetry with C-N  $1.393(10)$ , P-N  $1.697(7)$  Å, and N-P-N  $99.5(4)^\circ$ . Molecules of II have approximate  $C_{2v}$  symmetry, the  $N_2P-N-PN_2$  conformation being twisted slightly from eclipsed P lone-pair electrons. Mean bond lengths and angles in II are: Ph(H)N-P  $1.70(1)$ , PhN-P  $1.67(1)$ , C-N  $1.41$  Å, (H)N-P-N(H)  $94.9(5)$ , (H)N-P-N  $105.3(6)$ , P-N-P  $117.7(5)$ , P-N(H)-C  $121.1(5)^\circ$ , and P-N-C  $124.9(5)^\circ$ .

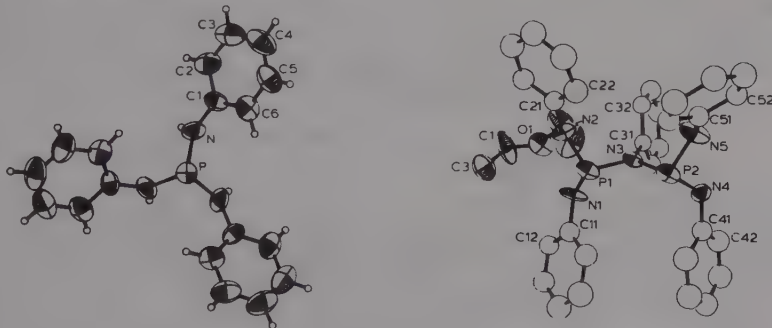


Fig. 1. The  $(C_6H_5NH)_3P$  (left) and  $[(C_6H_5NH)_2P]_2NC_6H_5$  (right) molecules.

# 2,4-BIS(DIMETHYLAMINO)-1,3-DIPHENYL-1,3-DIPHOSPHETANE

$C_{18}H_{24}N_2P_2$

G. BECKER, W. MASSA, O. MUNDT and R. SCHMIDT, 1982. *Z. Anorg. Chem.*, **485**, 23-35.

Monoclinic,  $P2_1/c$ ,  $a = 10.04$ ,  $b = 10.18$ ,  $c = 18.73$  Å,  $\beta = 105.15^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.035$  for 2289 reflexions (at  $-65^\circ C$ ).

The molecule contains two phenyl groups above and two dimethylamino groups below the four-membered diphosphetane ring. The ring is folded about the P...P line (dihedral angle between the two PCP planes is  $153^\circ$ ). The bond lengths and angles within the diphosphetane core (Fig. 1) are indicative of a significant degree of strain.



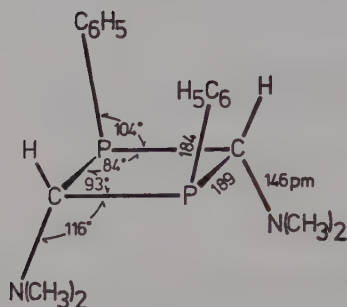


Fig. 1. Mean bond lengths and angles in  $C_{18}H_{24}N_2P_2$ .

1,2-DIPHENYL-3,3,4,4,5,5-HEXAMETHYL-1,2-DIPHOSPHACLOTTRISILANE  
 $C_{18}H_{28}P_2Si_3$

$(PhP)_2(SiMe_2)_3$

T.H. NEWMAN, J.C. CALABRESE, R.T. OAKLEY, D.A. STANISLAWSKI and R. WEST, 1982.  
 J. Organometal. Chem., **225**, 211-224.

Monoclinic,  $P2_1/c$ ,  $a = 12.526$ ,  $b = 11.828$ ,  $c = 15.412$  Å,  $\beta = 103.56^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.038$  for 3194 reflexions.

The molecule (Fig. 1) exists in the *dl(trans)* form with the five-membered ring significantly twisted. The angle between the P-P bond and the plane of the three Si atoms is  $42.9^\circ$ . The two Si-Si distances are equivalent (2.350 Å) and normal while the P-Si distances (2.287 and 2.268 Å) are rather long. The P-P distance is 2.204 Å. The ring angles at the P atoms (95.38 and 95.85°) are significantly smaller than those at the Si atoms (100.60, 100.78 and 101.61°).

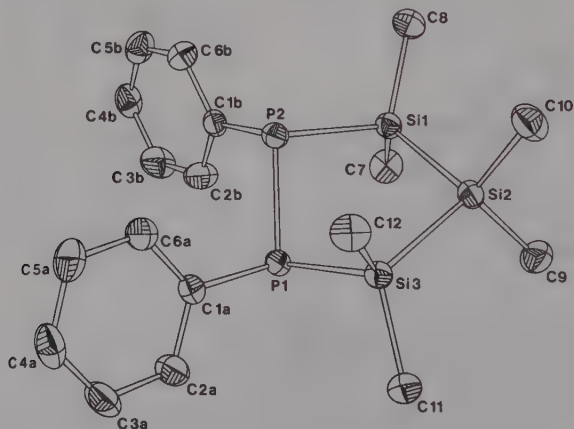


Fig. 1. Molecular structure of  $(PhP)_2(SiMe_2)_3$ .

1,2-DI-*t*-BUTYL-1,2-BIS(2,2-DIMETHYLPROPYL)DIPHOSPHANE-1,2-DIOXIDE

$C_{18}H_{40}O_2P_2$

$((C_4H_9)(C_5H_{11})PO)_2$

H. QUAST, M. HEUSCHMANN, W. VON DER SAAL, W. BUCHNER, K. PETERS and H.G. VON SCHNERING, 1982. Chem. Ber., 115, 1154-1161.

Orthorhombic,  $Fdd2$ ,  $a = 20.110$ ,  $b = 20.688$ ,  $c = 10.755$  Å,  $Z = 8$ . Mo radiation,  $R = 0.035$  for 1077 reflexions.

The centrosymmetric molecule (Fig. 1) assumes an approximately eclipsed conformation with the O atom and *t*-butyl group essentially diametrically opposed (OP-PC  $10.0^\circ$ ) due to the steric repulsion between the *t*-butyl ligands.

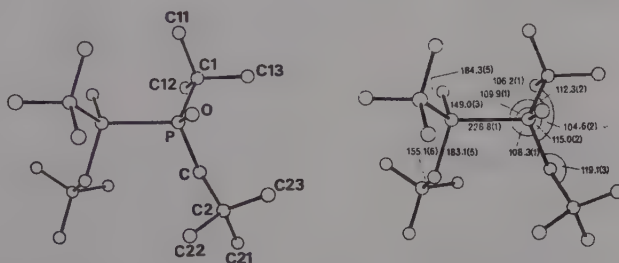


Fig. 1. The structure of  $((C_4H_9)(C_5H_{11})PO)_2$  with principal bond lengths (pm) and angles ( $^\circ$ ).

*o*-(DIPHENYLPHOSPHINO)BENZALDEHYDE

$C_{19}H_{15}OP$

$OHCC_6H_4P(C_6H_5)_2$

E.F. LANDVATTER and T.B. RAUCHFUSS, 1982. Organometallics, 1, 506-513.

Triclinic,  $P\bar{1}$ ,  $a = 10.663$ ,  $b = 11.065$ ,  $c = 8.585$  Å,  $\alpha = 103.20$ ,  $\beta = 105.95$ ,  $\gamma = 118.67^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.044$  for 2297 reflexions.

The gross structure of this tertiary phosphine (Fig. 1) resembles closely that of triphenylphosphine (1). The P-C distances are: P-C(1) 1.839, P-CA1 1.833, and P-CB1 1.838 Å and the angles about P are C(1)-P-CA1 102.3, C(1)-P-CB1 101.9 and CA1-P-CB1 102.0°. The C-C distances in the phenyl rings (A and B) range from 1.370 to 1.398 Å. Distances within the benzaldehyde moiety are: C(1)-C(2) 1.411, C(2)-C(3) 1.387, C(3)-C(4) 1.363, C(4)-C(5) 1.357, C(5)-C(6) 1.396, C(6)-C(1) 1.396, C(2)-C(7) 1.476 and C(7)-O 1.185 Å.

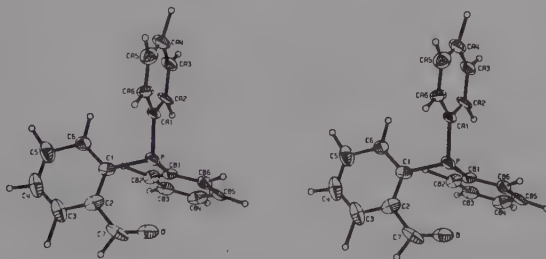
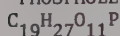


Fig. 1. Stereoview of the molecular structure of  $C_{19}H_{15}OP$ .

1. Structure Reports, 29, 638.

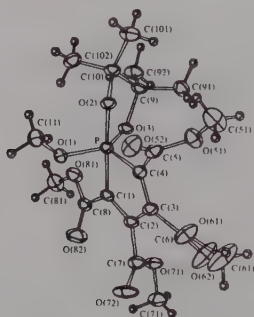
TETRAMETHYL 2-METHOXY-4,4,5,5-TETRAMETHYL-1,3,2-DIOXAPHOSPHOLIDINE-2-SPIRO-1'-  
PHOSPHOLE-2',3',4',5'-TETRACARBOXYLATE



I. BKOUCHE-WAKSMAN, P. L'HARIDON, Y. LEROUX and R. BURGADA, 1982. Acta Cryst., **B38**, 3024-3028.

Monoclinic,  $P2_1/c$ ,  $a = 13.908$ ,  $b = 8.231$ ,  $c = 19.624$  Å,  $\beta = 93.09^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.053$  for 2667 reflexions.

The P atom (Fig. 1) has trigonal bipyramidal coordination and lies  $0.074(1)$  Å from the  $O(1), O(3), C(4)$  plane.



P-O(1)	1.604 (2)
P-O(2)	1.659 (1)
P-O(3)	1.617 (1)
P-C(1)	1.885 (2)
P-C(4)	1.837 (2)

O(1)-P-O(3)	127.39 (9)
O(1)-P-C(4)	112.64 (9)
O(3)-P-C(4)	119.37 (9)
O(2)-P-O(1)	95.18 (8)
O(2)-P-O(3)	90.55 (7)
O(2)-P-C(4)	92.99 (9)
C(1)-P-O(1)	87.32 (9)
C(1)-P-O(3)	87.17 (8)
C(1)-P-C(4)	87.90 (10)
O(2)-P-C(1)	177.32 (8)

Fig. 1. The  $C_{19}H_{27}O_{11}P$  molecule and principal dimensions.

7-METHYL-12-ISOPROPYL-12-THIONO-7,12-DIHYDROBENZO-( $\alpha$ -PHENOPHOSPHAZINE)  
 $C_{20}H_{20}NPS$

V.M. IONOV, K.A. PASESHNICHENKO, V.B. RYBAKOV, I.B. ZASTENKER and L.A. ASLANOV, 1982. Zh. Strukt. Khim., **23**-4, 161-163 [J. Struct. Chem., **23**, 632-634].

Orthorhombic,  $P2_12_12_1$ ,  $a = 7.352$ ,  $b = 16.55$ ,  $c = 14.89$  Å,  $Z = 4$ . Mo radiation,  $R = 0.077$  for 978 reflexions.

The molecule (Fig. 1) contains planar A and B rings, the heterocyclic ring having a boat conformation with S equatorial. Distances involving P are: P-S 1.951(3), P-C<sub>1</sub> 1.779(9), P-C<sub>16</sub> 1.831(9), and P-C<sub>17</sub> 1.829(9) Å.

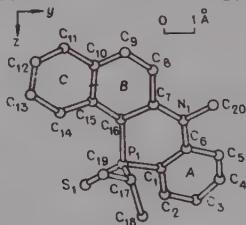
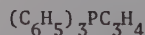


Fig. 1. The  $C_{20}H_{20}NPS$  molecule.

TRIPHENYLPHOSPHONIUM CYCLOPROPYLIDE



H. SCHMIDBAUR, A. SCHIER, B. MILEWSKI-MAHRLA and U. SCHUBERT, 1982. Chem. Ber., **115**, 722-731.

Hexagonal,  $P6_1$ ,  $a = 9.974$ ,  $c = 28.89$  Å,  $Z = 6$ . Mo radiation,  $R = 0.063$  for 1265 reflexions (at  $-20^\circ C$ ).

The cyclopropane ring and ylidic P-C bond are inclined by  $58^\circ$  to one another (Fig. 1) although the P-C bond length (1.696(6) Å) suggests significant multiple bond character.

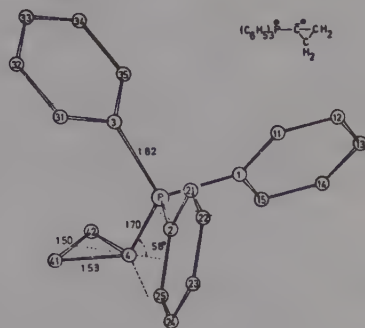


Fig. 1. Perspective view of  $(C_6H_5)_3PC_3H_4$  with principal bond lengths (pm).

7-BENZOYL-3-PHENYL-3-PHOSPHABICYCLO[3.3.1]NONAN-9-ONE 3-OXIDE HYDRATE  
 $C_{21}H_{21}O_3P, H_2O$  (I)

7-BENZOYL-3-PHENYL-3-PHOSPHABICYCLO[3.3.1]NONAN-9-ONE 3-OXIDE ETHANOL SOLVATE  
 $C_{21}H_{21}O_3P, C_2H_6O$  (II)

6-exo-HYDROXY-1,2-DIPHENYL-1-PHOSPHONIATRICYCLO[3.3.1.1<sup>3,7</sup>]DECANE IODIDE ETHANOL SOLVATE  
 $C_{21}H_{23}IOP, C_2H_6O$  (III)

H.J. MEEUWISSEN, G. SIRKS, F. BICKELHAUPT, C.H. STAM and A.L. SPEK, 1982. Rec. J.R. Neth. Chem. Soc., 101, 443-450.

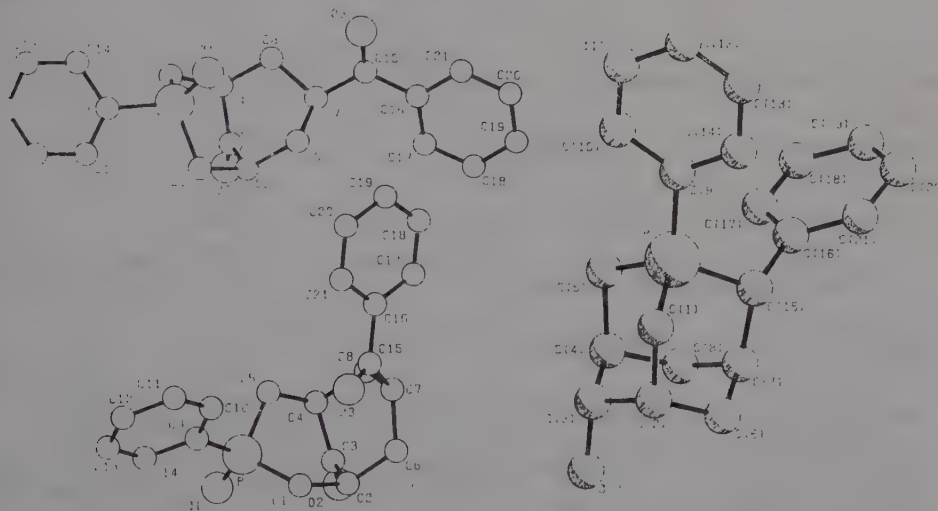


Fig. 1. Views of the two isomeric  $C_{21}H_{21}O_3P$  molecules (left) and of III (right).

I. Triclinic,  $P\bar{1}$ ,  $a = 10.534$ ,  $b = 10.648$ ,  $c = 9.651$  Å,  $\alpha = 91.74$ ,  $\beta = 110.05$ ,  $\gamma = 68.03^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.040$  for 2834 reflexions.

II. Triclinic,  $P\bar{1}$ ,  $a = 10.074$ ,  $b = 12.592$ ,  $c = 8.663$  Å,  $\alpha = 93.66$ ,  $\beta = 96.01$ ,  $\gamma = 66.53^\circ$ ,  $Z = 2$ . Cu radiation,  $R = 0.045$  for 2506 reflexions.

III. Triclinic,  $P\bar{1}$ ,  $a = 9.072$ ,  $b = 9.721$ ,  $c = 13.581$  Å,  $\alpha = 69.28$ ,  $\beta = 77.10$ ,  $\gamma = 65.22^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.05$  for 4727 reflexions.

The X-ray analyses revealed the configuration and conformation for I, II and III as shown in Fig. 1. In (III) the mean P-C bond length is 1.78 Å.

# 2-ACETYL-1-(DIPHENYLPHOSPHINYL)-4-METHYLCYCLOHEX-4-ENE

$C_{21}H_{23}O_2P$

S.D. DARLING and S.J. BRANDES, 1982. J. Org. Chem., 47, 1413-1416.

Monoclinic,  $P2_1/C$ ,  $a = 15.423$ ,  $b = 13.042$ ,  $c = 9.142$  Å,  $\beta = 96.16^\circ$ ,  $D_m = 1.216$ ,  $Z = 4$ . Mo radiation,  $R = 0.035$  for 1645 reflexions.

The analysis established the structure as shown in Fig. 1.

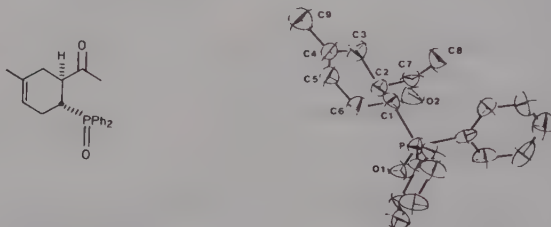


Fig. 1. The  $C_{21}H_{23}O_2P$  molecule.

# 2-METHYL-2-(TRIPHENYLPHOSPHONIO)DITHIOPROPIONATE

$C_{22}H_{21}PS_2$  (I)

# 3,5-DIISOPROPYLIDENE-1,2,4-TRITHIOLANE

$C_8H_{12}S_3$  (II)

U. KUNZE, R. MERKEL and W. WINTER, 1982. Chem. Ber., 115, 3653-3662.

I. Orthorhombic,  $P2_12_12_1$ ,  $a = 8.724$ ,  $b = 13.839$ ,  $c = 15.873$  Å,  $Z = 4$ . Mo radiation,  $R = 0.063$  for 2073 reflexions (at  $-100^\circ C$ ).

II. Monoclinic,  $C2/c$ ,  $a = 12.658$ ,  $b = 6.482$ ,  $c = 12.891$  Å,  $\beta = 109.01^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.053$  for 652 reflexions.

Molecule I (Fig. 1) exists in the open betaine form with a weak electrostatic interaction between P and S(1) ( $P \cdots S$  3.247(3) Å) and a markedly elongated P-C(2) bond (1.880(3) Å). Other major bond lengths are: C(2)-C(1) 1.526(4), C(1)-S(1,2) 1.679(3), P-C(phenyl) 1.809(3) Å. Torsion angles of interest are:  $P(1)C(2)-C(1)S(1)$   $-40.9(2)$ ,  $C(17)P(1)-C(2)C(3)$   $98.9(2)$ ,  $C(17)P(1)-C(2)C(4)$   $-18.8(2)^\circ$ . Molecule II (Fig. 1) has  $C_2$  symmetry with a half-chair ring conformation. Principal bond lengths are: S(1)-C 1.769(5), S(2)-C 1.752(6), S(2)-S(2') 2.057(3), C(1)-C(2) 1.348(7) Å.

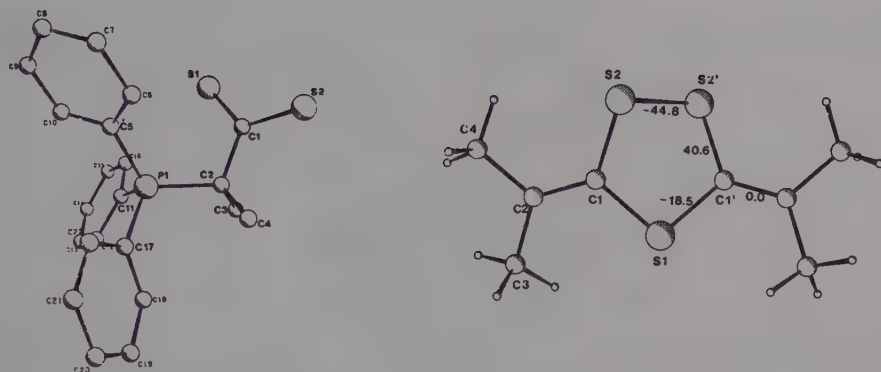


Fig. 1. Views of  $C_{22}H_{21}PS_2$  (I) (left) and  $C_8H_{12}S_3$  (II) (right) with torsion angles ( $^\circ$ ).

2,3-BENZO-6,7-DIMETHYL-8-PHENYL-4,8-BIS(TRIMETHYLSILYL)-6,7-DIAZA-1,5-DIPHOSPHABICYCLO[3.2.1]OCTANE

$C_{22}H_{34}N_2P_2Si_2$

R. APPEL, S. KORTE, M. HALSTENBERG and F. KNOCH, 1982. Chem. Ber., 115, 3610-3617.

Monoclinic,  $P2_1/c$ ,  $a = 18.466$ ,  $b = 10.706$ ,  $c = 13.247$  Å,  $\beta = 106.95^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.075$  for 3855 reflexions.

The structure was confirmed as that shown in Fig. 1.

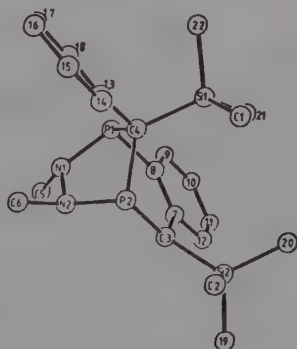


Fig. 1. The structure of  $C_{22}H_{34}N_2P_2Si_2$ .

1,2,3,4-TETRA-*t*-BUTYL-1,4-BIS(TRIMETHYLSILYL)TETRAPHOSPHANE

$C_{22}H_{54}P_4Si_2$

K.-F. TEBBE and R. FROHLICH, 1982. Z. Naturforsch., 37b, 534-541.

Monoclinic,  $P2_1/n$ ,  $a = 8.899$ ,  $b = 22.928$ ,  $c = 15.680$  Å,  $\beta = 94.14^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.042$  for 3220 reflexions.

The molecule (Fig. 1) forms a twisted Si-P<sub>4</sub>-Si chain with two distinct P-P bonds. Those involving a P bonded to Si are significantly shorter (2.188(1) Å) than the central P-P bond (2.214(1) Å). The mean P-Si length is 2.285(2) Å. There is a short intramolecular P(2)...P(5) contact (3.456(1) Å) which gives a nearly planar trapezium shape to the P<sub>4</sub> moiety.



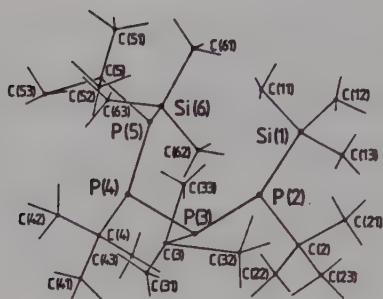
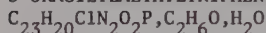


Fig. 1. Schematic representation of  $C_{22}H_{54}P_4Si_2$ .

5-URACILYMETHYLTRIPHENYLPHOSPHONIUM CHLORIDE ETHANOL HYDRATE



T. KOVACS and L. PARKANYI, 1982. Cryst. Struct. Comm., 11, 1565-1570.

Triclinic,  $P\bar{1}$ ,  $a = 9.994$ ,  $b = 10.298$ ,  $c = 13.435$  Å,  $\alpha = 100.80$ ,  $\beta = 103.46$ ,  $\gamma = 102.40^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.058$  for 3323 reflexions.

The cations (Fig. 1) form hydrogen-bonded dimers ( $N(3)-H \dots O(2)$ ,  $N \dots O$  2.870(4) Å). Main bond lengths are  $P-C(sp^2)$  1.792(3),  $P-C(sp^3)$  1.814(3) Å.

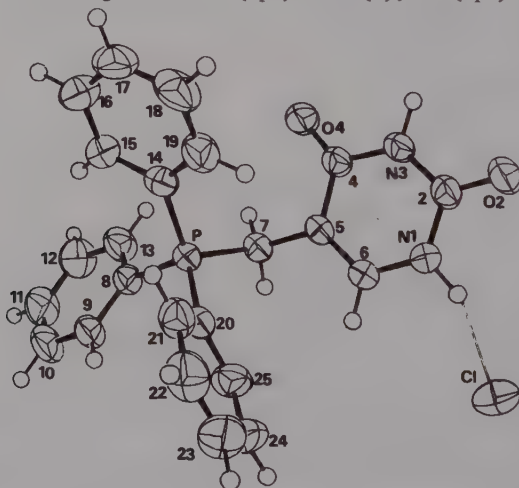
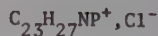
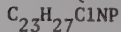


Fig. 1. A view of  $C_{23}H_{20}ClN_2O_2P$ .

BENZYL(DIETHYLAMINO)DIPHENYLPHOSPHONIUM CHLORIDE

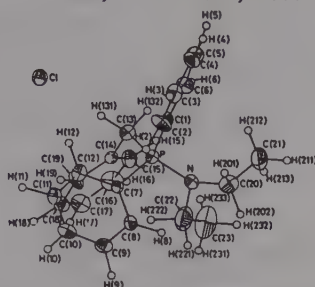


J.C.J. BART, I.W. BASSI and M. CALCATERRA, 1982. Acta Cryst., B38, 1932-1936.

Monoclinic,  $P2_1/c$ ,  $a = 12.515$ ,  $b = 10.327$ ,  $c = 16.052$  Å,  $\beta = 94.83^\circ$ ,  $D_m = 1.21$ ,  $Z = 4$ . Mo radiation,  $R = 0.082$  for 956 reflexions.

Phosphorus is tetrahedrally coordinated (Fig. 1) with mean  $P-C(sp^2)$  1.80(1) and  $P-C(sp^3)$  1.76(1) Å. The  $P^+-N$  distance is 1.63(1) Å. The  $P-C(sp^2)$  and  $P-N$  bonds are in a slightly distorted, staggered conformation (gauche-, gauche+ and trans) with

respect to the  $C(sp^2)-CH_2$  bond of the benzyl residue. The diethylamino group shows disorder. Packing is by van der Waals interactions with shortest non-bonded contacts  $Cl...C$  3.77,  $Cl...H$  2.70,  $C...C$  3.48,  $C...H$  2.58,  $H...H$  2.13 Å.



II. Triclinic,  $P\bar{1}$ ,  $a = 10.691$ ,  $b = 10.851$ ,  $c = 11.184$  Å,  $\alpha = 64.57$ ,  $\beta = 75.19$ ,  $\gamma = 80.88^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.030$  for 3342 reflexions.

The N atom of I (Fig. 1) is situated on an inversion centre and neighbouring molecules are joined by short symmetrical O-H...O hydrogen bonds (2.392(4) Å). Principal bond lengths and angles are: P-N 1.535(1), P-C 1.794(2), P-O 1.519(2) Å; P-N-P 180, P-O-H 124.1(2)°. In II the O atoms have been replaced by S atoms and the molecule exists in a different tautomeric form (Fig. 1). Principal bond lengths and angles are: P-N 1.677(2), P-C 1.805(2), P-S 1.943(1) Å; P-N-P 132.6(1), S-P-N 115.1(1)°.

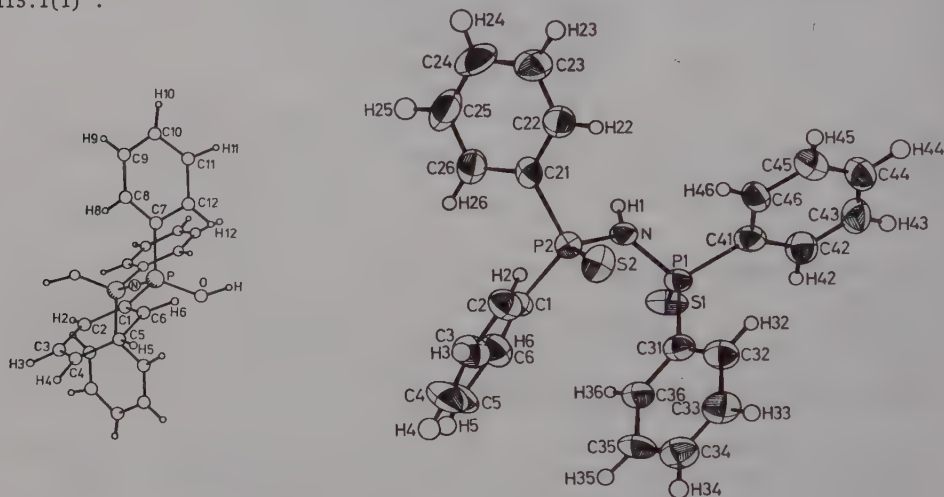


Fig. 1. The structures of  $C_{24}H_{21}NO_2P_2$  (I) (left) and  $C_{24}H_{21}NP_2S_2$  (II) (right).

10-CHLORO-9,10-DIHYDRO-1,2',9-TRIMETHYL-3,5',7-TRIPHENYLSPIRO[1H-[1,3,2λ<sup>5</sup>,4λ<sup>5</sup>]DIAZA-DIPHOSPHETO[2,1-c:2,3-c']BIS[1,2,4,3λ<sup>5</sup>]TRIAZAPHOSPHOLE-5,3'-[2H][1,2,4,3λ<sup>5</sup>]TRIAZAPHOSPHOLE]

$C_{24}H_{24}ClN_9P_2$

$[C_6H_5CN_3(CH_3)]_3P_2Cl$

R.O. DAY, A. SCHMIDPETER and R.R. HOLMES, 1982. *Inorg. Chem.*, **21**, 3916-3919.

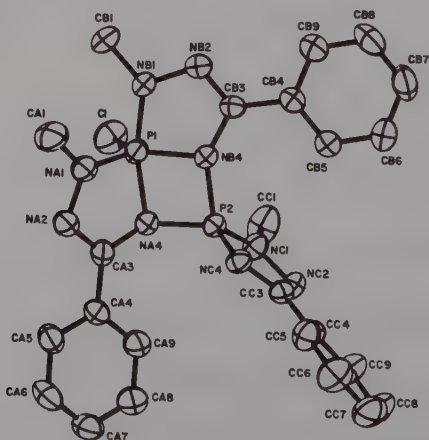


Fig. 1. The  $C_{24}H_{24}ClN_9P_2$  molecule.

Monoclinic,  $C2/c$ ,  $a = 33.229$ ,  $b = 9.685$ ,  $c = 17.677$  Å,  $\beta = 111.96^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.035$  for 2881 reflexions.

The molecule (Fig. 1) contains both four- and five-coordinate P atoms incorporated in the phosphetidine ring with attached 1,2,4,3-triazaphosphole rings. A partial meridional, partial cis-facial ring arrangement exists about the penta-coordinated P atom which has a geometry intermediate between trigonal bipyramidal and square pyramidal (C1 apical). Bond lengths: P1-C1 2.094(1), P1-NA1 1.656(3), P1-NB1 1.704(3), P1-NB4 1.705(2), P1-NA4 1.790(2), P2-NA4 1.669(2), P2-NB4 1.688(2), P2-NC1 1.628(2), and P2-NC4 1.577(2) Å.

2,2',3,3',4,4'-HEXA(t-BUTYL)-1,1'-BICYCLOTETRAPHOSPHANE  
 $C_{24}H_{54}P_8$

M. FEHER, R. FROHLICH and K.-F. TEBBE, 1982. *Z. Krist.*, **158**, 241-253.

Monoclinic,  $P2_1/n$ ,  $a = 11.416$ ,  $b = 18.057$ ,  $c = 17.648$  Å,  $\beta = 108.32^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.076$  for 5361 reflexions.

The molecule (Fig. 1) with approximate symmetry  $C_{2h}$  consists of two  $\sigma$ -bonded, trans positioned and slightly folded four-membered rings of phosphorus atoms. The exocyclic bridge between the two rings with P-P 2.218 Å is comparable with the mean endocyclic P-P bond length (2.21 Å) although the individual values in both rings are slightly different. The t-butyl substituents are in trans-arrangement. The mean P-C (1.890 Å) and C-C (1.524 Å) are as expected.

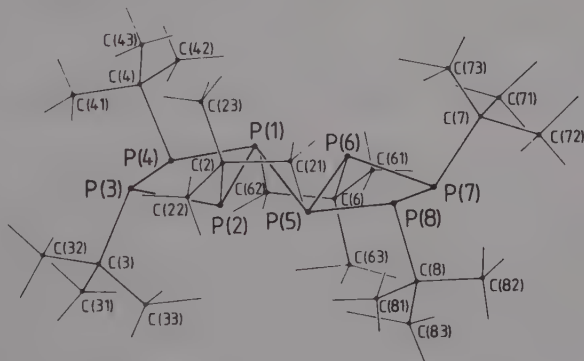


Fig. 1. The  $C_{24}H_{54}P_8$  molecule.

1-(2'-TRIPHENYLPHOSPHONIO-3',3',4',4'-TETRAFLUOROCYCLOBUTENYL) (3,3-DIFLUOROCYCLOBUTENE-2,4-DIONATE) CHLOROFORM SOLVATE  
 $C_{26}H_{15}F_6O_2P \cdot 0.5(CHCl_3)$

F.W.B. EINSTEIN and T. JONES, 1982. *Canad. J. Chem.*, **60**, 2065-2068.

Monoclinic,  $C2/c$ ,  $a = 29.624$ ,  $b = 10.224$ ,  $c = 18.576$  Å,  $\beta = 116.26^\circ$ ,  $Z = 8$ . Mo radiation,  $R = 0.042$  for 1652 reflexions.

The compound is a zwitterion (Fig. 1) consisting of a phosphonium cation and a delocalized anion. Principal dimensions are: P-C 1.773-1.786(5) Å, C-P-C 106.4-111.7(2)° in the cation fragment, and C-C 1.388-1.440(8), C-O 1.216 and 1.208(7) Å in the anionic fragment.

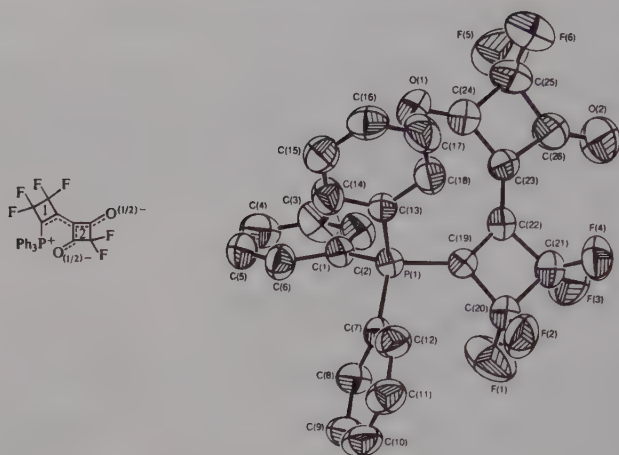


Fig. 1. The zwitterion structure of  $C_{26}H_{15}F_6O_2P$  and an ORTEP diagram.

(R,S)-DIPHENYL 1-(4-BROMOANILINO)-1-(4,5-METHYLENEDIOXY-2-NITROPHENYL)METHANE-  
PHOSPHONATE HEMI-BENZENE SOLVATE

$C_{26}H_{20}BrN_2O_7P \cdot 0.5(C_6H_6)$

M.D. CRENSHAW, S.J. SCHMOLKA, H. ZIMMER, R. WHITTLE and R.C. ELDER, 1982. J. Org. Chem., 47, 101-104.

Monoclinic,  $P2_1/n$ ,  $a = 10.779$ ,  $b = 20.694$ ,  $c = 13.336$  Å,  $\beta = 109.470^\circ$ ,  $D_m = 1.48$ ,  $Z = 4$ . Mo radiation,  $R = 0.054$  for 2968 reflexions.

The X-ray structure of the phosphonate (Fig. 1) indicates that there is steric hindrance about the protocarbanionic C-atom C(1); the nitro group in the ortho-position lies above this atom, effectively completing a cage around it. The nitro group is rotated  $34.5^\circ$  from the plane of the phenyl ring giving a  $O(21) \cdots H(1)$  distance of 2.15 Å and  $C(1)-H(1) \cdots O(21)$   $128^\circ$ . Although the carbanion is formed on addition of base, the subsequent nucleophilic attack on (e.g.) aldehydes is prevented. The molecular dimensions are compared with those of a related phosphonate, (R,S)-diethyl-1-anilino-1-phenylmethanephosphonate (1) (which does react with aldehydes) and are found to be very similar except in the remote ester groups.

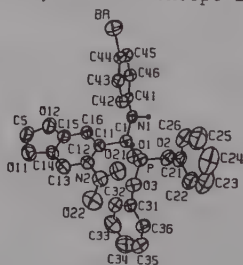


Fig. 1. A molecule of  $C_{26}H_{20}BrN_2O_7P$ .

1. Structure Reports, 47B, 626.

3,3a-DIHYDRO-2,3,3-TRIPHENYL-2H-CYCLOHEPT[d]-1,2-OXAPHOSPHOLE-2-OXIDE  
 $C_{26}H_{21}O_2P$  (I)

3,3a-DIHYDRO-2,3,3,3a,4,5,6-HEPTAPHENYL-2H-CYCLOPENT[d]-1,2-OXAPHOSPHOLE-2-OXIDE  
 $C_{48}H_{35}O_2P$  (II)

G. MAAS, M. REGITZ, K. URGAST, M. HUFNAGEL and H. ECKES, 1982. Chem. Ber., 115, 669-682.

I. Monoclinic,  $P2_1/c$ ,  $a = 13.450$ ,  $b = 9.636$ ,  $c = 15.765$  Å,  $\beta = 92.25^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.036$  for 1856 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 11.040$ ,  $b = 23.880$ ,  $c = 15.160$  Å,  $\beta = 114.90^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.045$  for 1382 reflexions.

The structures of I and II were confirmed as those shown above. The conformation of the seven-membered rings are similar and are characterized by the structural parameters  $\alpha$  ( $47.9^\circ$  (I),  $42.2^\circ$  (II)) and  $\beta$  ( $24.4^\circ$  (I),  $23.0^\circ$  (II)). The five-membered rings adopt different conformations: in I an envelope with P at the flap; in II a half-chair.

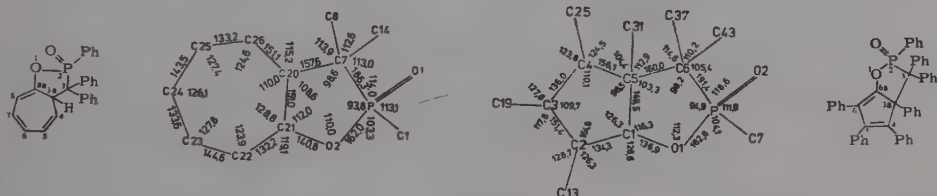


Fig. 1. Bond lengths (pm) and angles ( $^\circ$ ) in the bicyclo moieties of  $C_{26}H_{21}O_2P$  (left) and  $C_{48}H_{35}O_2P$  (right). The phenyl groups are omitted for clarity.

trans-2,4-DITHIO-2,4-BIS(N-METHYLANILINO)-1,3-DIPHENYL-1,3,2,4-DIAZADIPHOSPHETIDINE  
 $C_{26}H_{26}N_4P_2S_2$  (I) trans- $[C_6H_5N(CH_3)P(S)NC_6H_5]_2$

trans-2,4-DITHIO-2,4-BIS(N-ETHYLANILINO)-1,3-DIPHENYL-1,3,2,4-DIAZADIPHOSPHETIDINE  
 $C_{28}H_{30}N_4P_2S_2$  (II) trans- $[C_6H_5N(C_2H_5)P(S)NC_6H_5]_2$

D.E. COONS, V.S. ALLURED, M.D. NOIROT, R.C. HALTIWANGER and A.D. NORMAN, 1982. Inorg. Chem., 21, 1947-1952.

I. Triclinic,  $P\bar{1}$ ,  $a = 10.131$ ,  $b = 10.043$ ,  $c = 13.524$  Å,  $\alpha = 90.16^\circ$ ,  $\beta = 108.75^\circ$ ,  $\gamma = 102.86^\circ$ ,  $D_m = 1.365$ ,  $Z = 2$ . Mo radiation,  $R = 0.034$  for 2611 reflexions.

II. Monoclinic,  $P2_1/n$ ,  $a = 10.280$ ,  $b = 19.059$ ,  $c = 7.531$  Å,  $\beta = 112.10^\circ$ ,  $D_m = 1.325$ ,  $Z = 2$ . Mo radiation,  $R = 0.033$  for 1282 reflexions.

Both structures consist of centrosymmetric molecules (Fig. 1), that of I containing two crystallographically independent molecules. Mean structural parameters for I are: P-N(ring) 1.690(2), P-N(exo) 1.636(2), P-S 1.914(1) Å, P-N-P(ring) 98.3(1), N-P-N(ring) 81.7(1), N-P-N(exo) 109.6(1), and S-P-N(exo) 113.7(1) $^\circ$ . Corresponding parameters for II are: 1.690(4), 1.632(3), 1.913(1) Å, 98.6(1), 81.4(1), 109.8(8), and 113.8(1) $^\circ$ .



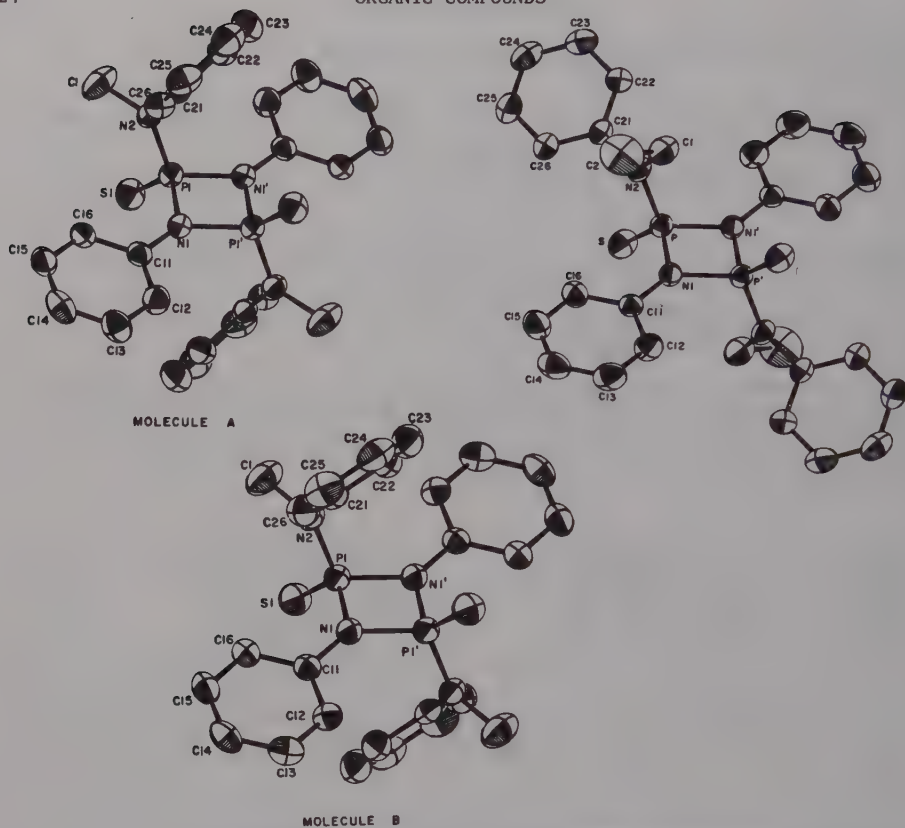


Fig. 1. The two independent molecules of  $\text{trans-[C}_6\text{H}_5\text{N(CH}_3\text{)P(S)NC}_6\text{H}_5\text{]}_2$  (left) and the  $\text{trans-[C}_6\text{H}_5\text{N(C}_2\text{H}_5\text{)P(S)NC}_6\text{H}_5\text{]}_2$  molecule (right).

2,3-DIPHENYL-4-(PHENYLPHOSPHINIDENE)-2-(TRIMETHYLSILOXY)-1-(TRIMETHYLSILYL)-1,3-AZAPHOSPHETIDINE  
 $\text{C}_{26}\text{H}_{33}\text{NOP}_2\text{Si}_2$

R. APPEL, M. HALSTENBERG, F. KNOCH and H. KUNZE, 1982. *Chem. Ber.*, **115**, 2371-2373.

Monoclinic,  $\text{P2}_1/\text{c}$ ,  $a = 10.739$ ,  $b = 11.822$ ,  $c = 22.117 \text{ \AA}$ ,  $\beta = 93.01^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.054$  for 6442 reflexions.

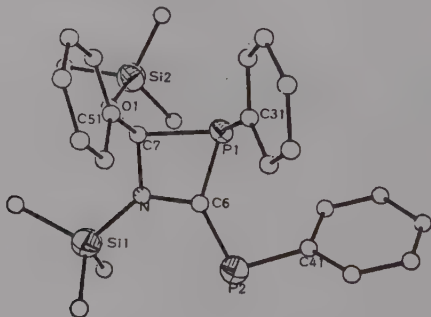


Fig. 1. The structure of  $\text{C}_{26}\text{H}_{33}\text{NOP}_2\text{Si}_2$ .

The structure was established as that shown in Fig. 1. Principal bond lengths are: P(1)-C(6) 1.84(1), P(1)-C(7) 1.94(1), P(2)-C(6) 1.70(1), N-C(6) 1.38(1), N-C(7) 1.48(1), N-Si(1) 1.78(1), O(1)-Si(2) 1.65(1) Å.

TETRAPHENYLPHOSPHONIUM BIS(TRIFLUOROMETHYLSULFONYLIMIDO)ETHOXYsulphinate

$C_{28}H_{25}F_6N_2O_5PS_3$  (I)

$(C_6H_5)_4P^+, (CF_3SO_2N)_2S(OC_2H_5)^-$

BIS(TETRAPHENYLPHOSPHONIUM) TRIS(PHENYLSULFONYLIMIDO)SULPHITE

$C_{66}H_{55}N_3O_6P_2S_4$  (II)

$2(C_6H_5)_4P^+, (C_6H_5SO_2N)_3S^{2-}$

H.W. ROESKY, W. SCHMIEDER, W. ISENBERG, W.S. SHELDRIK and G.M. SHELDRIK, 1982. Chem. Ber., 115, 2714-2727.

I. Monoclinic,  $P2_1/c$ ,  $a = 8.979$ ,  $b = 11.337$ ,  $c = 31.881$  Å,  $\beta = 97.05^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.064$  for 2568 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 35.526$ ,  $b = 13.203$ ,  $c = 12.430$  Å,  $\beta = 97.56^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.082$  for 4111 reflexions.

In I (Fig. 1) the anion adopts a trans, trans configuration and the central S atom exhibits trigonal-pyramidal coordination. Principal bond lengths are: S(3)-O(5) 1.591(3), S(3)-N 1.609(4), N(1,2)-S(1,2) 1.575(4) Å. In II (Fig. 1) the central S atom again exhibits trigonal-pyramidal coordination; potential  $C_3$  symmetry is not realized because only two  $C_6H_5SO_2$  groups are in a cis configuration. Principal bond lengths are: S(4)-N 1.61(1), N(1,2,3)-S(1,2,3) 1.56(1) Å.

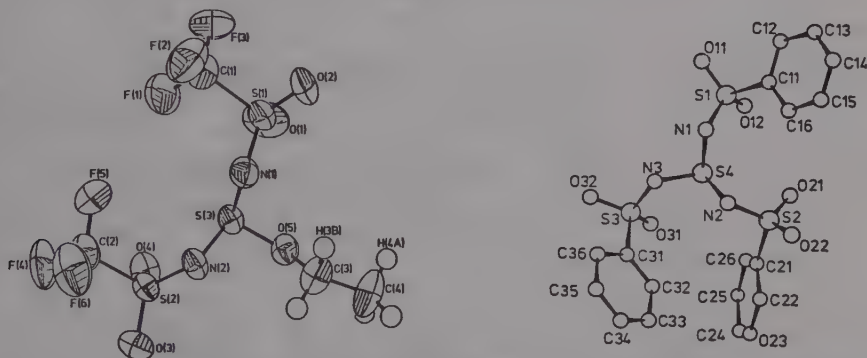


Fig. 1. The structure of the  $(CF_3SO_2N)_2S(OC_2H_5)^-$  and  $(C_6H_5SO_2N)_3S^{2-}$  anions.

2,8-DICHLORO-3,7-DI-(2-HYDROXYETHOXY)-5-METHOXY-4,6-DIPHENYLDIBENZOPHOSPHOLE 5-OXIDE  
 $C_{29}H_{25}Cl_2O_6P$  (I)

5-HYDROXY-3,7-DIMETHOXY-4,6-DI-(4-METHOXY-3,5-DI-t-BUTYLPHENYL)DIBENZOPHOSPHOLE  
 5-OXIDE DIETHYL ETHER SOLVATE

$C_{44}H_{57}O_6P, C_4H_{10}O$  (II)

J. CORNFORTH, D.D. RIDLEY, A.F. SIERAKOWSKI, D. UGUEN, T.W. WALLACE and P.B. HITCHCOCK, 1982. J. Chem. Soc. Perkin I, 2317-2331.

I. Monoclinic,  $P2_1/c$ ,  $a = 14.716$ ,  $b = 13.839$ ,  $c = 15.502$  Å,  $\beta = 120.01^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.087$  for 1789 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 14.303$ ,  $b = 14.942$ ,  $c = 22.311$  Å,  $\beta = 103.22^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.065$  for 3895 reflexions.

The bond lengths in the benzo rings of I (Fig. 1) are not significantly different from normal, and the P-C bonds are equal, P-C(1) 1.82(1) and P-C(2) 1.81(2) Å. The P-O bonds are significantly different with P-O(1) 1.49(1) and P-O(4) 1.57(1) Å being respectively double and single bonds. The dibenzophosphole nucleus is slightly bowed about the line joining the P atom to the mid point of the C(6)-C(20) bond. The P atom is 0.08 Å above the mean plane of the three rings and C(2) and C(6) are 0.06 Å below it. The pendant phenyl groups are slightly bent towards the P atom at C(7) and C(21), with the angle between the C(2)-C(7) and C(16)-C(21) bonds 45°, while that between the lines C(7)...C(10) and C(21)...C(24) is only 38°. The only short intermolecular contact is between O(1) and O(3) of a related molecule which at 2.79 Å suggests hydrogen bonding. In II (Fig. 2) the dibenzophosphole nucleus bond lengths are similar to those in I, with P-C bond lengths of 1.808(4) and 1.816(4) and P-O double and single bonds 1.465(3) and 1.568(4) Å respectively. There is again bowing of this fragment with an angle of 3° between the two outer ring mean planes. The pendant phenyl rings are distorted due to steric crowding, being bent along C(7)...C(10) and C(19)...C(22). The convex side of the former points towards the centre of the molecule while in the latter the concave side faces the centre. The distortion also results in a reduction of the ring angle to 116.3° (mean) at the t-butyl substituents, elongation of the ring bonds to C(10) and C(22) to 1.404(8) (mean) and longer than normal bonds to the substituents with C(ring)-C(t-butyl) 1.542(8) (mean) and C(ring)-O(Me) 1.401(2) Å. There is a hydrogen bond from O(1)H to O(7) of the diethyl ether solvate with O(1)...O(7) 2.61 and H(1)...O(7) 1.95 Å. There are no other short contacts in the crystal packing of II.

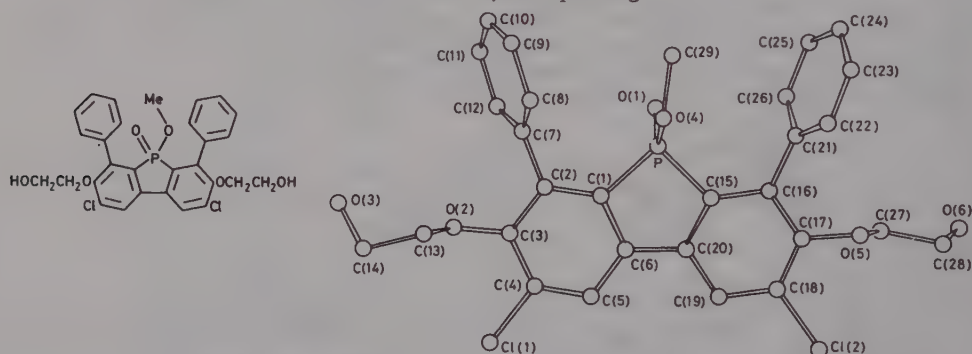


Fig. 1. Molecular skeleton and structure of  $C_{29}H_{25}Cl_2O_6P$  (I).

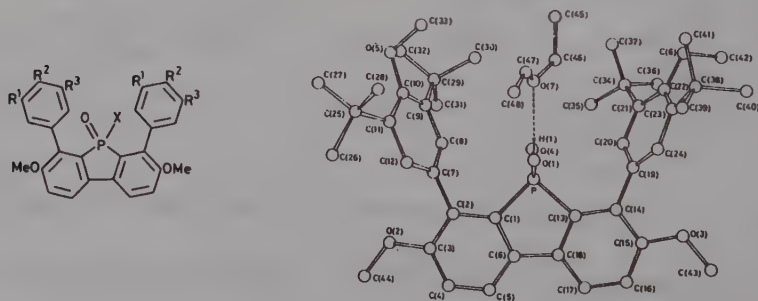


Fig. 2. Molecular skeleton ( $R^1 = R^3 = \text{But}^t$ ,  $R^2 = \text{OMe}$ ,  $X = \text{OH}$ ) and structure of  $C_{44}H_{57}O_6P, C_4H_{10}O$  (II).

[BIS-(2,6-DIMETHYLPHENYL)THIOPHOSPHORO]-N,N'-DICYCLOHEXYLSOTHIURONIUM TETRAFLUORO-

BORATE

$C_{29}H_{42}BF_4N_2O_2PS_2$

$C_{29}H_{42}N_2O_2PS_2^+ \cdot BF_4^-$

C. BLONSKI, M.-B. GASC, A. KLAÉBÉ, J.-J. PÉRIÉ, R. ROQUES, J.P. DECLERCQ and G. GERMAIN, 1982. *J. Chem. Soc. Perkin II*, 7-13.

Monoclinic,  $P2_1/a$ ,  $a = 15.802$ ,  $b = 13.409$ ,  $c = 15.050$  Å,  $\beta = 96.82^\circ$ ,  $Z = 4$ .  $R = 0.055$  for 3144 reflexions.

Bonds C(6)-N(7) and C(6)-N(8) are equal within experimental error (mean 1.308 Å) confirming the isouronium structure (Fig. 1). The P(1)-O(4) and P(1)-O(5) bonds are also equal (mean 1.581 Å), the P(1)-S(2) double bond is 1.898 and the P(1)-S(3) single bond is 2.128 Å. H(7), N(7), C(6), N(8) and H(8) lie in a plane with H(7)-N(7) and H(8)-N(8) antiperiplanar. This plane lies at an angle of  $77^\circ$  to that of C(6)-S(3)-P(1) leading to N(7)...P(1) and N(8)...P(1) distances of 3.896 and 3.490 Å respectively.

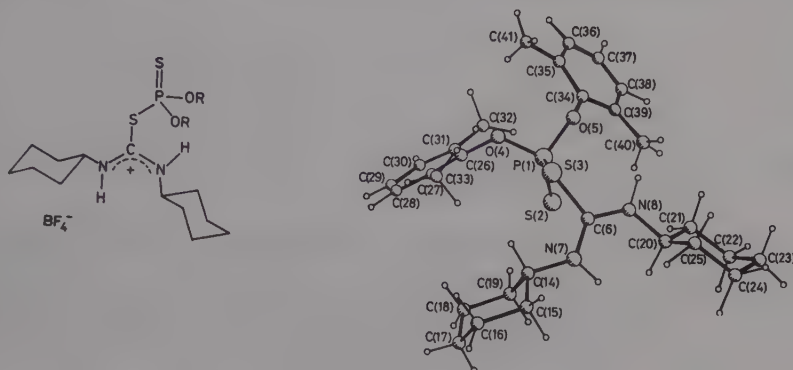


Fig. 1. Molecular skeleton and structure of  $C_{29}H_{42}N_2O_2PS_2^+$ .

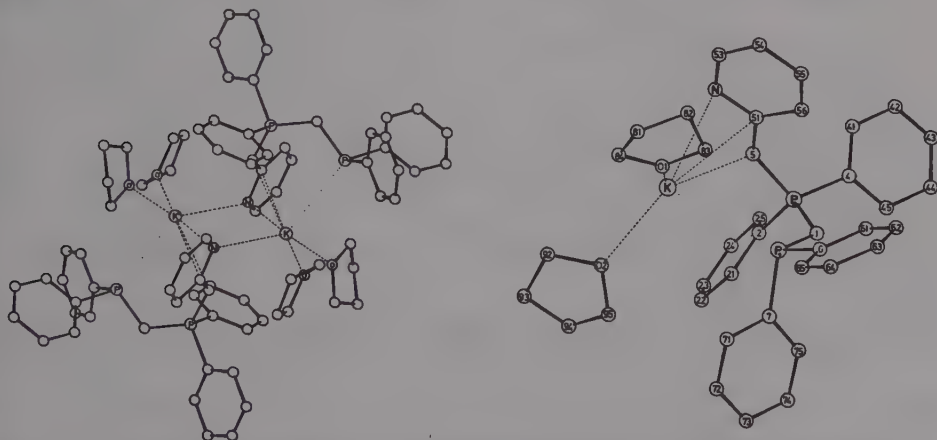
POTASSIUM DIPHENYLPHOSPHONIUM(DIPHENYLPHOSPHINOMETHYLIDE) -2-PYRIDINYLMETHYLIDE BIS(TETRAHYDROFURAN)

$C_{31}H_{26}KNP_2 \cdot 2(C_4H_8O)$

$K^+, C_{31}H_{26}NP_2^-, 2(C_4H_8O)$

H. SCHMIDBAUR, U. DESCHLER and B. MILEWSKI-MAHRLA, 1982. Chem. Ber., **115**, 3290-3299.

Monoclinic,  $P2_1/n$ ,  $a = 14.231$ ,  $b = 19.413$ ,  $c = 14.217$  Å,  $\beta = 114.17^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.106$  for 3356 reflexions (at  $-20^\circ C$ ).



lengths involving P are: P(1)-C(1) 1.752(8), C(7) 1.802(11), C(6) 1.846(8); P(2)-C(1) 1.689(8), C(2) 1.827(8), C(4) 1.840(8), C(5) 1.691(9) Å.

1,3-DIPHENYL-6'-(PHENYLETHENYLIDENE)SPIRO(1-BENZOPHOSPHORINE-2(1H),1'-CYCLOHEXA-2',4'-DIENE)-1-OXIDE  
 $C_{34}H_{25}OP$  (I)

PHENYL-BIS(2-(trans-2-PHENYLETHENYL)PHENYL)PHOSPHANE  
 $C_{34}H_{27}P$  (II)

T. BUTTERS, I. HALLER-PAULS and W. WINTER, 1982. Chem. Ber., 115, 578-592.

I. Monoclinic,  $P2_1/c$ ,  $a = 8.737$ ,  $b = 18.17$ ,  $c = 16.398$  Å,  $\beta = 104.16^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.043$  for 2413 reflexions.

II. Monoclinic,  $P2_1/c$ ,  $a = 9.660$ ,  $b = 17.497$ ,  $c = 17.322$  Å,  $\beta = 117.28^\circ$ ,  $Z = 4$ . Cu radiation,  $R = 0.059$  for 3048 reflexions.

In I (Fig. 1) the heterocyclic ring adopts a half-chair conformation with P(1) and C(21) displaced from the C(7),C(12),C(13),C(14) mean plane by -0.38 and 0.34 Å respectively. The dihedral angle between the mean planes of the two rings of the spiro junction is  $80.0^\circ$ . Bond lengths and angles have expected values. In II the P coordination is trigonal pyramidal; bond lengths and angles have expected values. The two trans stilbene moieties are not planar; the two phenyl rings in each are rotated by  $36.1$  and  $21.3^\circ$  with respect to one another.

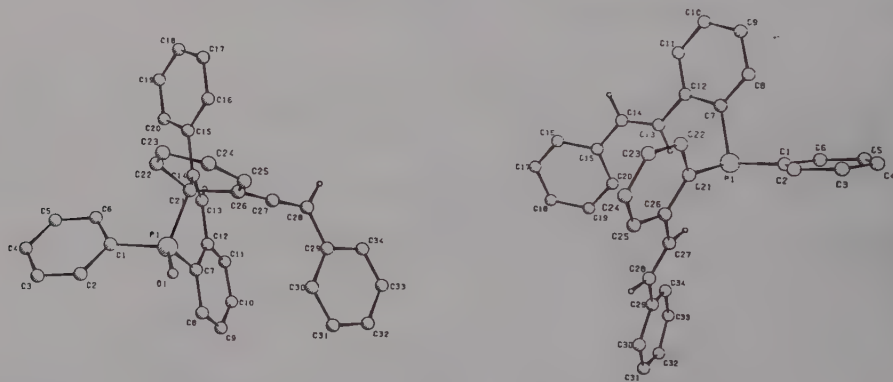


Fig. 1. Perspective views of  $C_{34}H_{25}OP$  (left) and of  $C_{34}H_{27}P$  (right).

TRIETHYLAMMONIUM 5-HYDROXY-3,3,7,7-TETRAPHENYL-1,4,6,9-TETRAOXA-5-PHOSPHASPIRO[4.4]-NONANE-2,8-DIONE  
 $C_{34}H_{36}NO_7P$

A. DUBOURG, R. ROQUES, G. GERMAIN, J.-P. DECLERCQ, B. GARRIGUES, D. BOYER, A. MUÑOZ, A. KLAËBÉ and M. COMTAT, 1982. J. Chem. Research, S, 180-181; M, 1901-1948.

Triclinic,  $P\bar{1}$ ,  $a = 11.192$ ,  $b = 11.155$ ,  $c = 13.830$  Å,  $\alpha = 75.22$ ,  $\beta = 89.29$ ,  $\gamma = 113.62^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.10$ .

The structure of the hydroxyphosphorane anion is that of a slightly deformed trigonal bipyramid with the equatorial plane O(2),O(6),O(10) containing P(1) (Fig. 1). The P-O<sup>-</sup> distance is 1.47 Å. Other P-O distances are: P(1)-O(3) 1.77, P(1)-O(6) 1.61, P(1)-O(7) 1.78, P(1)-O(10) 1.61 Å. The dioxaphospholan rings are quasi planar and slightly twisted.



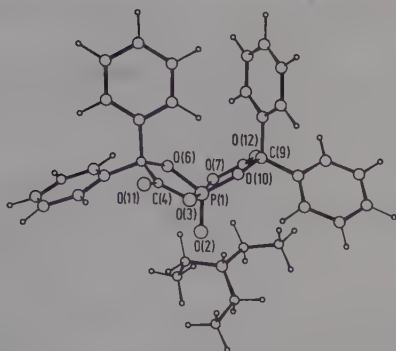


Fig. 1.  $C_{34}H_{36}NO_7P$ : the molecular structure.

1-1-DIPHENYL-3-ETHOXY- $\lambda^5$ - $\sigma^6$ -PHOSPHAINDOLE-YLIDENE-CYCLOPHOSPHORANE FLUORENONE ADDUCT  
 $C_{35}H_{27}O_2P$

R. BOHME and E. WILHELM, 1982. Cryst. Struct. Comm., 11, 7-11.

Monoclinic,  $P2_1$ ,  $a = 9.944$ ,  $b = 14.108$ ,  $c = 10.362$  Å,  $\beta = 114.74^\circ$ ,  $D_m = 1.27$ ,  $Z = 2$ .  
 Mo radiation,  $R = 0.067$  for 1922 reflexions.

The geometry around the phosphorous atom (Fig. 1) is that of a distorted trigonal bipyramid. The molecular structure and the reactions of the compound are consistent with the postulates about the mechanism of the Wittig reaction.

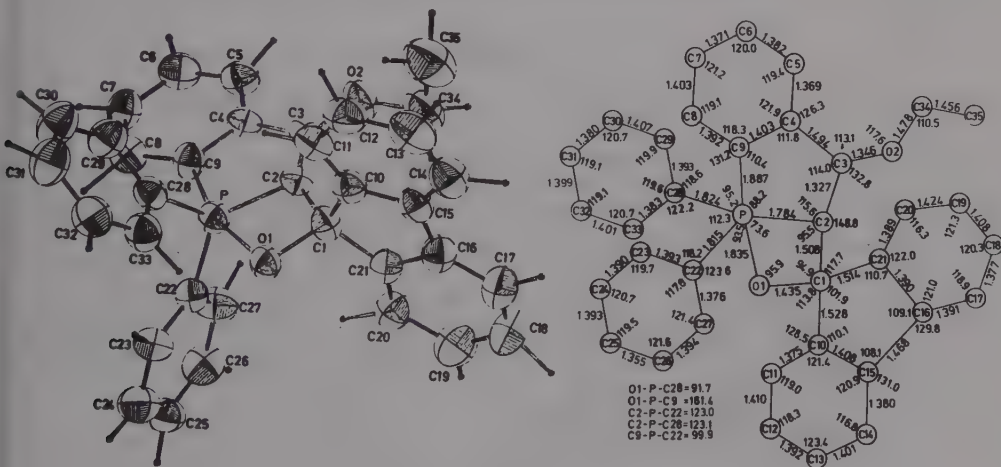


Fig. 1. The  $C_{35}H_{27}O_2P$  molecule and dimensions.

meso-1,1'-(1,2-ETHANEDIYL)BIS(1,2,3,4-TETRAHYDRO-4,4-DIMETHYL-1-PHENYLPHOSPHINOLIUM)  
 DIPERCHLORATE

$C_{36}H_{42}Cl_2O_8P_2$

$[C_{36}H_{42}P_2]^{2+}(ClO_4^-)_2$

N. GURUSAMY, K.D. BERLIN, D. van der HELM and M.B. HOSSAIN, 1982. J. Am. Chem. Soc., 104, 3107-3114.



Monoclinic,  $P2_1/c$ ,  $a = 10.4905$ ,  $b = 21.694$ ,  $c = 16.571 \text{ \AA}$ ,  $\beta = 105.53^\circ$ ,  $D_m = 1.337$ ,  $Z = 4$ . Cu radiation,  $R = 0.054$  for 5056 reflexions.

The molecule (Fig. 1) assumes a near anti conformation with a deviation of C-P dihedral angles from  $180$  to  $143.6^\circ$  and a slightly flattened ring containing the P atom. The relative configuration of the substituents at P(1) and P(1)' are quite different.

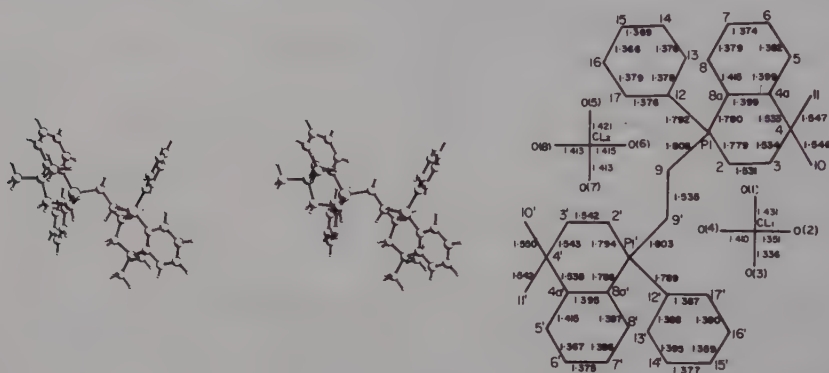


Fig. 1. Stereoview of the cation (left) and bond lengths ( $\sigma = 0.003$ - $0.004$  for P-C and C1-O,  $0.004$ - $0.007 \text{ \AA}$  for C-C) in  $[C_{36}H_{42}P_2]^{2+}(C_{104}^-)_2$ .

TRIPHENYL(P,P,P-TRIPHENYLPHOSPHINE-IMIDATO-N)PHOSPHORUS(I) THIOCYANATE

$C_{37}H_{30}N_2P_2S$

$(Ph_3P)_2N^+, SCN^-$

C. GLIDEWELL and H.D. HOLDEN, 1982. J. Organometal. Chem., 226, 171-181.

Monoclinic,  $P2_1/c$ ,  $a = 10.836$ ,  $b = 12.744$ ,  $c = 24.051 \text{ \AA}$ ,  $\beta = 103.33^\circ$ ,  $Z = 4$ . Mo radiation,  $R = 0.13$  for 4627 reflexions.

The structure consists of cations  $(Ph_3P)_2N^+$  which are markedly non-linear at N (P-N-P  $136.3^\circ$ ), and anions  $SCN^-$  (which exhibit both positional and orientational disorder) (Fig. 1). The N-P distances are  $1.592$  and  $1.579 \text{ \AA}$ , the P-C distances range from  $1.79$  to  $1.82 \text{ \AA}$ , and the P...P distance is  $2.941 \text{ \AA}$ . In the anion, main dimensions are: S-C  $1.74$  and  $1.86$ , C-N  $1.50$  and  $1.39 \text{ \AA}$ , C-S-C  $83^\circ$ , S-C-N  $162$  and  $160^\circ$ .

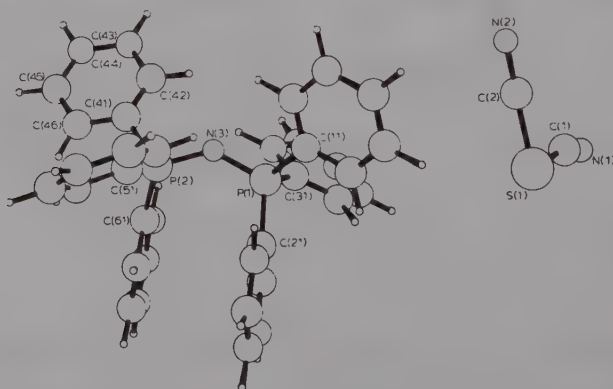


Fig. 1. The molecular structure of  $(Ph_3P)_2N^+, [SCN]^-$ .

2,2'-BIS(O-(OXO-DIPHENYLPHOSPHINO)PHENYLOXY)DIETHYL ETHER DICHLOROMETHANE SOLVATE  
 $C_{40}H_{36}O_5P_2 \cdot CH_2Cl_2$

A.L. SPEK, E.T. DE RUITER and W.M.G.F. PONTENAGEL, 1982. *Cryst. Struct. Comm.*, **11**, 1869-1872.

Tetragonal,  $P\bar{4}2_1c$ ,  $a = 15.30$ ,  $c = 16.55$  Å,  $D_m = 1.26$ ,  $Z = 4$ . Cu radiation,  $R = 0.053$  for 1908 reflexions.

The ether (Fig. 1) has twofold crystallographic symmetry. The P-O(3) distance is 1.485(3) Å.

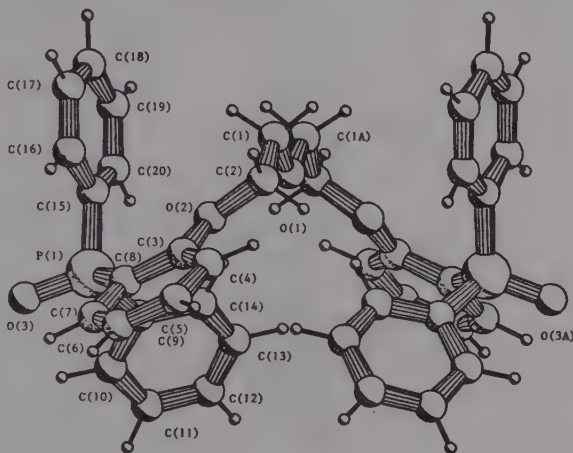


Fig. 1. A view of  $C_{40}H_{36}O_5P_2$ .

1,1,1-TRIS[(DIPHENYLPHOSPHINO)METHYL]ETHANE (TRIPHOS)  
 $C_{41}H_{39}P_3$

C. MEALLI, 1982. *Acta Cryst.*, **B38**, 1040-1043.

Triclinic,  $P\bar{1}$ ,  $a = 13.487$ ,  $b = 13.573$ ,  $c = 10.484$  Å,  $\alpha = 104.73$ ,  $\beta = 106.37$ ,  $\gamma = 97.73^\circ$ ,  $D_m = 1.19$ ,  $Z = 2$ . Mo radiation,  $R = 0.077$  for 3073 reflexions.

The three-armed molecule (Fig. 1) has neither crystallographic nor pseudo threefold symmetry as the conformation of one of the three aliphatic chains markedly differs from that of the other two; some disorder is apparent in the arm centred on P(3). Bond distances and angles are normal and there are no unusually short intermolecular contacts.

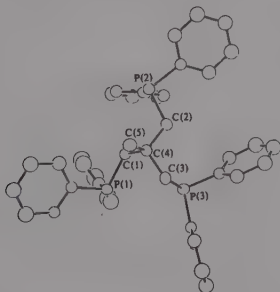
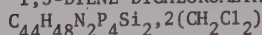


Fig. 1. A view of the  $C_{41}H_{39}P_3$  molecule.

meso-1,3,4,6-TETRAPHENYL-2,5-BIS(PHENYL(TRIMETHYLSILYL)AMINO)-1,3,4,6-TETRAPHOSPHAHEXA-1,5-DIENE DICHLOROMETHANE SOLVATE



R. APPEL, V. BARTH and M. HALSTENBERG, 1982. Chem. Ber., **115**, 1617-1625.

Monoclinic,  $\text{P2}_1/\text{c}$ ,  $a = 10.755$ ,  $b = 21.476$ ,  $c = 13.579$  Å,  $\beta = 127.09^\circ$ ,  $Z = 2$ . Mo radiation,  $R = 0.078$  for 2104 reflexions (at  $-80^\circ\text{C}$ ).

The centrosymmetric molecule (Fig. 1) is in the meso-form with a trans arrangement at the P-P bond. The P-C double bonds have E,E-configuration. Principal bond lengths are: P-P 2.23(1), P(2)-C(1) 1.84(1), P(1)-C(1) 1.69(1) Å.

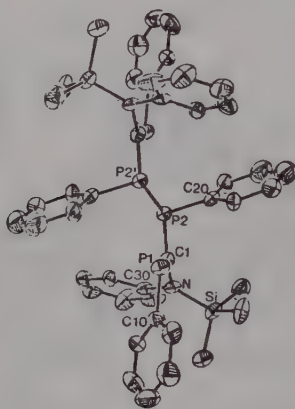
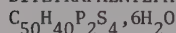


Fig. 1. The structure of  $\text{C}_{44}\text{H}_{48}\text{N}_2\text{P}_4\text{Si}_2$ .

DITETRAPHENYLPHOSPHONIUM TETRATHIOOXALATE HEXAHYDRATE



H. LUND, E. HOYER and R.G. HAZELL, 1982. Acta Chem. Scand., **B36**, 207-209.

Tetragonal,  $\text{P4}$ ,  $a = 13.026$ ,  $c = 13.786$  Å,  $Z = 2$ . Cu radiation,  $R = 0.062$  for 1370 reflexions.

The structure consists of an I-centred arrangement of the tetraphenylphosphonium ions in a cell with the c axis halved. The anions and water molecules are placed in holes in this lattice (Fig. 1). The tetrathiooxalate ion has crystallographic twofold symmetry but is non-planar, with a torsion angle of  $79.5^\circ$ . The C-S distances are 1.713 and 1.691 Å and the C-C' distance is 1.46 Å. The angles S(1)-C-S(2), S(1)-C-C' and S(2)-C-C' are respectively  $128.6^\circ$ ,  $114.7^\circ$  and  $116.6^\circ$ .

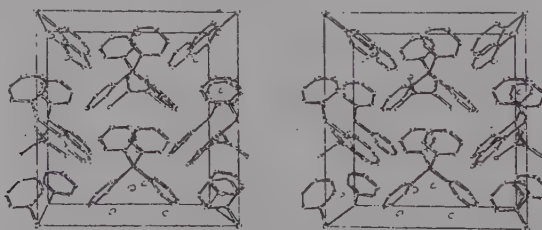


Fig. 1. Stereoscopic view of the crystal structure of  $\text{C}_{50}\text{H}_{40}\text{P}_2\text{S}_4 \cdot 6\text{H}_2\text{O}$ . Unconnected atoms are water molecules.













